

UNCLASSIFIED

AD NUMBER
AD865842
NEW LIMITATION CHANGE
TO Approved for public release, distribution unlimited
FROM Distribution: Further dissemination only as directed by Director, Advanced Research Projects Agency, Washington, DC 20301, Feb 1970 or higher DoD authority.
AUTHORITY
ARPA ltr, !8 Aug 1970

THIS PAGE IS UNCLASSIFIED

AD

OOD ENGINEERING REPORT NO. 41

OPTICAL MATERIALS STUDY PROGRAM

FINAL TECHNICAL REPORT

(Period Ending January 20, 1970)

The Perkin-Elmer Corporation, Norwalk, Connecticut

OPTICAL OPERATIONS DIVISION

WILLIAM R. GOGGIN, Program Manager

February, 1970

Sponsored by

Advanced Research Projects Agency

Department of Defense

ARPA Order No. 885

Contract No. DAAHO1-69-C-0950

This document may be further distributed by any holder only with specific prior approval of Director, Advanced Research Projects Agency, Washington, D.C. 20301.

O.O.D. ENGINEERING REPORT NO. 41

OPTICAL MATERIALS STUDY PROGRAM

FINAL TECHNICAL REPORT

(Period Ending January 20, 1970)

THE PERKIN-ELMER CORPORATION
OPTICAL OPERATIONS DIVISION
NORWALK, CONNECTICUT

PROGRAM MANAGER, WILLIAM R. GOGGIN

FEBRUARY 1970

SPONSORED BY

ADVANCED RESEARCH PROJECTS AGENCY
DEPARTMENT OF DEFENSE
ARPA ORDER NO. 885
CONTRACT NO. DAAH01-69-C-0950

This document may be further distributed by any holder only with specific prior approval of - Director, Advanced Research Projects Agency, Washington, D.C. 20301

CONTRIBUTORS TO REPORT

Perkin-Elmer Corporation

W. R. Goggin

R. A. Paquin

W. S. Poor

A. F. Slomba

Batelle Memorial Institute

R. E. Maringer

A. N. Ashurst

D. Hauser

M. J. Ryan

R. Shemanski

W. R. Goggin

Program Manager

Perkin-Elmer Corporation

TABLE OF CONTENTS

<u>SECTION</u>	<u>TITLE</u>	<u>PAGE</u>
1.0	INTRODUCTION	1
2.0	OBJECTIVE AND SCOPE	3
3.0	MATERIALS	6
4.0	MIRROR DEVELOPMENT AND OPTICAL CHARACTERIZATION	16
4.1	Polishing of Mirrors	16
4.1.1	Beryllium Mirrors	16
4.1.2	Dielectric Mirrors	18
4.2	Optical Characterization	19
4.2.1	Polished Bare Beryllium Surfaces	19
4.2.2	Anodized Beryllium Surfaces	19
4.2.3	Optical Coatings on Mirror Surfaces	25
5.0	TESTING AND MEASUREMENTS	28
5.1	General	28
5.2	The Precision Surface Interferometer	28
5.3	Real Time Dimensional Stability Measurements	34
5.4	The Dimensional Interferometer	36
5.5	Thermal Dimensional Stability	38
5.6	Homogeneity Evaluations	47
5.6.1	Thermal Expansion Coefficient Measurements	47
5.6.2	X-Ray Diffraction	47
5.7	Residual Stress Measurements	49
5.8	Radiation Damage	51
6.0	BATTELLE MEMORIAL INSTITUTE SUBCONTRACT	53
6.1	General	53
6.2	Task 1 - Characterization	54
6.3	Fabrication	58
6.3.1	Task 2 - Solid Mirror Blanks	58
6.3.2	Task 3 - Solid Beryllium Cubes	58
6.3.4	Task 4 - Lightweight Mirror Blanks	59
6.4	Task 5 - Joining Studies	59
6.4.1	Diffusion Bonding	59
6.4.1.1	Preparation of Preliminary Specimens	59
6.4.1.2	Preparation of Tensile Specimens	66
6.4.1.3	Preparation of Disc Specimens	68
6.4.2	Brazing	70
6.4.2.1	Preparation of Preliminary Specimens	71
6.4.2.2	Preparation of Tensile Specimens	74
6.4.2.3	Preparation of Disc Specimens	75
7.0	SUMMARY	76
	REFERENCES	79

LIST OF TABLES

<u>TABLE</u>	<u>TITLE</u>	<u>PAGE</u>
3-1	Hardness of Beryllium Samples	9
3-2	Thermal Properties of Mirror Materials	12
4-1	Perkin-Elmer Treatment of Beryllium Mirrors	20
4-2	Beryllium Anodizing Parameters	21
4-3	Total Hemispherically Scattered Energy	26
5-1	Measurement Data from Test Program VII	30
5-2	Measurement Data from Test Program VIII	31
5-3	Measurement Data from Test Program IX	32
5-4	Real Time Dimensional Stability	35
5-5	Linear Dimensional Stability Data from Precision Dimensional Interferometer	37
5-6	Thermal Dimensional Stability	39
5-7	Thermal Stability of Beryllium Mirrors Before and After Joining	41
5-8	Thermal Expansion Coefficients	48
6-1	Beryllium Samples Prepared by BMI	56
6-2	Tensile Strengths of Joined Beryllium Specimens	69
6-3	Brazing Experiments	72

LIST OF ILLUSTRATIONS

	PAGE
3-1 Thermal Dependence of the Coefficient of Thermal Expansion	14
3-2 Dependence of Thermal Conductivity on Temperature	15
4-1 Reflectance of Polished Beryllium Bare and Anodized	23
5-1 Thermal Stability of Sample No. 199 Before and After Brazing	43
5-2 Thermal Stability of Sample No. 200 Before and After Diffusion Bonding	44
5-3 Thermal Stability of Sample No. 201 Before and After Diffusion Bonding	45
5-4 Radiation Effects on Beryllium Surfaces	52
6-1 Machined Arch Lightweight Mirror Design	60
6-2 Diffusion Bonded Nickel Coated Beryllium	62
6-3 Diffusion Bonded Silver Coated Beryllium	64
6-4 Diffusion Bonded Silver Coated Beryllium	65
6-5 Diffusion Bonded Nickel Coated Beryllium	67
6-6 Diffusion Bonded Silver Coated Beryllium	67

ABSTRACT

Various materials are being investigated for use as diffraction limited mirror substrates. Included are dielectrics, such as fused silica, low expansion glasses and glass ceramics, and beryllium (Be) based materials. Material and processing specifications for Be optical components are developed and recommended. Sectioned Be mirrors were brazed and diffusion bonded and successfully polished and tested. Low reflectivity and very low scatter are reported for an anodized optically polished bare Be surface. Results of test programs of Precision Surface Interferometer are presented and discussed. Most dielectrics are temporally and thermally stable to approximately the limit of detectability. Hydrostatically prepared Be is more stable than the commercial counterparts and the reattritioning process has provided further improvement. The role of residual stress in thermal and temporal instabilities is presented. Data from Linear Dimensional Interferometer and Vacuum Dilatometer are presented. Details of brazing and diffusion bonding experiments for Be are included.

FOREWORD

This Research was supported by the
ADVANCED RESEARCH PROJECTS AGENCY, DEPARTMENT OF
DEFENSE, and was monitored by the U.S. ARMY MISSILE
COMMAND under Contract No. DAAH01-69-C-0950.

The findings of this report are not
to be construed as an official Department of the Army
position unless so designated by other authorized
documents.

Destroy this report when no longer needed.
Do not return it to the originator.

SECTION 1.0

INTRODUCTION

Requirements of high acuity optical mirror systems necessitate the use of optical components of extremely high dimensional stability. This stability must be maintained over the design mirror lifetime and over a range of service environments.

The choice of mirror substrate materials is limited due to stringent optical surface geometry and stability requirements. To date no single substance has proven entirely suitable over the complete spectrum of service environments.

Mirror stability and optical performance are closely controlled and limited by several factors. These include material type, method of manufacture and optical processing, expected service lifetime, and the influence of environmental effects.

The Optical Materials Study Program is a research and development project designed to determine whether selected materials have, or can be made to have the requisite stability for high acuity optical mirror applications. Materials under investigation include traditional fused silica as well as others which show outstanding promise because of attractive physical or mechanical properties.

A major portion of this effort is directed toward developing improved materials and techniques for fabricating beryllium. Consequently, a number of Be compositions produced by various techniques are being investigated. Beryllium products are being fabricated by Battelle Memorial Institute (BMI), Columbus ,

Ohio, under subcontract* from this program. In addition, beryllium compacts prepared by commercial vendors, and Stanford Research Institute (SRI), Menlo Park, California, are also being tested.

This is the Final Technical Report of Contract No. DAA H01-69-C-0950. Details are given on accomplishments to date in mirror fabrication, quality control, optical characteristics, structural homogeneity, and dimensional stability testing.

* Perkin-Elmer Subcontract No. 48052-UD to Battelle Memorial Institute, R. E. Maringer, Program Manager

SECTION 2.0

OBJECTIVE AND SCOPE

The OBJECTIVE of the Optical Materials Study Program is to evaluate candidate materials for high performance optical mirror applications, and to augment their development wherever possible to achieve a given set of operational goals. These goals, simply stated, are the following for a diffraction limited application:

1. A given mirror material must be capable of being fabricated in the sizes and quantities required.
2. A mirror material must be polishable to high surface quality (with respect to figure accuracy, scatter, etc.) and be a receptive host to one or more types of special purpose optical coatings.
3. A mirror material must be polishable to within $\lambda/50$ *rms of its designed surface geometry, and remain stable within this requirement over its expected service life and range of environmental conditions.

Some of the above requirements, especially those relating to potential service environments, are difficult to satisfy with any one material. In addition, the choice of candidate mirror materials is definitely limited. Through this program then, the above requirements are being investigated in detail for a number of potentially stable optical mirror materials.

Total optical performance of a mirror is not solely determined by the intrinsic material characteristics, but also by behavioral perturbations imposed by methods of manufacture,

* λ = wavelength

mechanical and optical processing, and the service environment. The SCOPE of this program includes inquiring into each of these influences, and is therefore concerned with mechanical, metallurgical, chemical, and optical characteristics of mirror materials.

Materials being evaluated include fused silica, low expanding dielectrics such as CER-VIT* and ULE**, beryllium-based metals, silicon, TZM, beryllium oxide, aluminum, and others.

A major effort is being directed toward development of improved beryllium-based mirror materials. Beryllium products from the major commercial domestic suppliers as well as those produced by several exploratory processes are under study. These latter include beryllium billets manufactured by the hot isostatic pressing process of Battelle Memorial Institute, and the pressureless sintering process of Stanford Research Institute.

Research activities on beryllium mirror materials at BMI are supported under subcontract from this program. In addition to development of a stable beryllium substrate, feasibility tests are being conducted on diffusion-bonding and brazing methods to enable the construction of large mosaic mirror structures from small component parts.

The following report summarizes program activities from 21 January 1969 to 20 January 1970. Previous reports issued under this program are listed on the following page.

* CER-VIT, registered trademark of Owens-Illinois

**ULE, registered trademark of Corning Glass Works

PREVIOUS REPORTS OF
THE OPTICAL MATERIALS STUDY PROGRAM

<u>PERKIN-ELMER REPORT NO.</u>	<u>DATE</u>	<u>DDC* CAT. NO.</u>
8845	June 1966	AD-485577-L
8545	Oct. 1966	AD-800551-L
8656	Jan. 1967	AD-805205-L
8903	Aug. 1967	AD-819941-L
8997	Oct. 1967	AD-825047-L
9158	Dec. 1967	AD-836890-L
9254	March 1968	AD-830705-L
9300	June 1968	
9383	Sept. 1968	AD-842356-L
OOD. NO. 10	May 1969	AD-853936-L
OOD. NO. 13	Aug. 1969	AD-858201-L
OOD. NC. 29	Nov. 1969	

* DDC - Defense Documentation Center

SECTION 3.0

MATERIALS

At present, over 250 mirror test samples remain within our inventory. Of these, 151 are beryllium and include specimens of 18 different material types, 6 manufacturers and 5 fabrication methods.

In addition to quantitative evaluations of all specimens, specific emphasis is being placed on the qualitative performance of beryllium products produced by the various manufacturing methods. To achieve this comparison, solid beryllium cylindrical discs produced by the hot isostatic pressing process⁽¹⁾, pressureless-sintering⁽²⁾, and the commercial process of vacuum hot pressing are undergoing similar test schedules. To assist in this evaluation, a number of mirror samples of the three processes were made from the same material type and lot. This evaluation was conducted on the following three powder types:

- a. P-40* (a high oxide, fine-grained instrument grade quality powder)
- b. P-50* (a medium oxide, fine-grained, high strength Be-Cr alloy powder)
- c. SP* (a recently developed, low oxide, fine-grained beryllium powder)

* Powder designations of Kawecki-Berylco Industries

The fine-grained powders are used as starting materials to achieve improved homogeneity since an individual crystallite grain of beryllium is intrinsically anisotropic. Structures made from relatively coarse grained powder exhibit a great part of this basic anisotropy. By using a very fine-grained powder, a more isotropic structure is achieved by statistically randomizing the orientation of a great number of individual grains in a given volume. Powder grain sizes used in earlier phases of this study program were approximately 75 μm . The P-40, P-50, and SP types listed above have grain sizes of 6, 13, and 8 μm , respectively. A modified powder handling treatment (called reattritioning) for increasing homogeneity in beryllium⁽³⁾ has been very successful as evidenced by results described in Section 5.0 of this report. This technique was used by BMI to produce beryllium samples. A complete description of this process is given in Section 6.2 of the second quarterly report issued under this contract.⁽⁴⁾

Detailed intercomparison of beryllium materials and manufacturing processes are discussed in Section 5.0. In generalizing these results, it can be said that the hydrostatic methods of fabrication (the SRI and BMI methods) yield products which are superior in performance to those produced by commercial methods.

At the present time, fully stabilized beryllium mirrors are stable to better than $\lambda/50$ rms per year, and $\lambda/50$ rms over a temperature change of 50°C.

From a materials point of view, all of the three material types considered can be produced in a form with high temporal and

thermal stability. Optical characterization of these materials (see Section 4.2) indicates that the P-50 and SP products are noticeably superior. On the other hand the P-40 material, because of its high oxide and poor sintering characteristics, cannot be conveniently processed to full density. It therefore has poor reflectivity and optical scatter characteristics. The P-50 and SP materials can be prepared to near full density and have optical scatter approaching premium-grade glass optics. The utilization of P-50, SP or equivalent grade materials is therefore recommended for high quality mirror substrate materials.

Knoop hardness values for several beryllium samples are listed in Table 3-1. Within a given material type the BMI samples have greater hardness than the commercial products (KBI, BBC) and the SRI produced materials. This higher hardness is most probably related to work hardening during processing and is apparently not related to any fundamental difference in chemistry. This work hardening is related to stresses applied during pressing. The low processing temperatures for BMI and KBI (900°C and 1000°C respectively) require mechanical pressures to achieve full consolidation of powder, whereas the SRI process uses high temperature (1200°C) and no applied pressures.

The SRI process, because of the high processing temperature cannot develop the high strength potential of the Be-Cr alloy (P-50). Strength of pressureless sintered P-50 is quite low⁽²⁾ and not noticeably different from that of the other unalloyed SRI materials.

TABLE 3-1

HARDNESS OF BERYLLIUM SAMPLES

Material Type	Hardness According to Fabricator* Knoop (500 gm load)			
	KBI	BBC	SRI	EMI
P-50	240-250		190-200	335-341
P-40/1-400	225-295	225-235	185-195	360-400
SP	200-220		200-210	250-260
S-200		185-200		240-250
Alloyed Be			250-265	

*KBI Kowacki-Beryllco Industries, Inc.
 BBC Brush Beryllium Company
 SRI Stanford Research Institute
 EMI Battelle Memorial Institute

Conversely, the BMI process has resulted in higher P-50 strengths than those achieved in a vacuum hot pressed product. (5)

Strength however, is not the most important criterion for mirrors. Dimensional stability is the paramount consideration and cannot be sacrificed for additional strength in high precision optical components. Alloying elements have been added in the manufacture of several SRI mirror blanks with no large differences in dimensional stabilities. However, the strength of the alloy mirrors is significantly increased over the unalloyed beryllium. (6)

A number of the traditional oxide-based dielectric materials which form glassy or glass-ceramic solids, are also under investigation. Some of the glassy solids are:

- fused silica (Corning, General Electric Co.)
- ULE (Corning)
- Z-Glass (Heraeus-Schott)
- ZEROSIL (Heraeus-Schott)

The glass-ceramics are identified as:

- CER-VIT (Owens-Illinois Glass)
- OHARAX (Ohara Optical Glass Mfg. Co.)
- GLAS-KERAMIK (Heraeus-Schott)
- Corning-9623 (Corning)
- Corning-9622 (Corning)

In a previous report, (7) examples of internal structural defects such as bubbles, striations and inclusions typical to some of these materials were shown. It was also shown that the polishability of a surface was not affected by striations intersecting that surface. Since glasses polish primarily by a plastic flow polishing mechanism, many defects can be buried by a thin flowed glass surface

layer. These defects are generally not visible from a surface, even when using high magnification electron-microscopy. By chemically etching an optical surface many of these defects become apparent because of localized differences in electrochemical potential. Electron micrographs of polished and polished-and-etched mirror surfaces on ULE and CER-VIT were shown. (7)

In order to achieve stable optical performance in environments subject to thermal transients or gradients, it is necessary to have mirrors whose optical figures are independent of temperature changes or gradients. Several figures of merit, based on a material's physical properties, have been used to describe deformations due to thermal effects. Several of these merit indicators are shown in Table 3-2 along with their numerical values (at 20°C) for several candidate mirror materials. Relative rankings for these indicators are also given.

Two figures of merit are often used to compare thermal deformation characteristics of materials. One of these is expressed by the ratio $(K/\alpha\rho)$ where (K) is the thermal conductivity, (α) the thermal expansion coefficient and (ρ) is the density. A high value of this ratio signifies a minimum of dimensional change due to thermal expansion. Comparison of materials by this indicator may not be universally valid as it expresses dilation (or contraction) under generally isothermal or slowly varying thermal conditions. A better figure of merit is the thermal diffusivity $(D = K/\rho C)$ where (C) is the specific heat and the other terms are as previously stated. A high diffusivity is desirable since the lifetime of a thermal transient is inversely proportional to (D) and the magnitude of a thermal distortion is proportional to (α/D) .

TABLE 3-2

THERMAL PROPERTIES OF MIRROR MATERIALS

Material	$\frac{K}{\rho \alpha}$ ($10^3 \text{ cal-cm}^2/\text{gm-sec}$)	$D = (K/\rho C)$ (cm^2/sec)	$\frac{\alpha}{D}$ ($10^{-6} \text{ sec}/^\circ\text{C} - \text{cm}^2$)
Beryllium	17. (4)	0.42	27. (5)
Beryllium Oxide	27. (2)	0.70	9.5 (3)
Fused Silica	2.7 (7)	0.01	68. (8)
ULE	23. (3)	0.01	8.1 (2)
CER-VIT	16. (5)	0.01	13. (4)
Silicon	56. (1)	0.80	2.9 (1)
Aluminum (2014)	5.7 (6)	0.57	40. (6)
Titanium (6Al - 4V)	1.4 (9)	0.08	99. (9)
Invar	2.6 (8)	0.03	48. (7)

In general, an athermalized mirror material should have an infinitely large thermal conductivity and zero thermal expansion coefficient. Unfortunately no real material exists with this combination of properties.

The quantities shown in Table 3-2 are temperature dependent and the listed data are only valid for operation near 20°C. To illustrate this, Figure 3-1 shows the thermal dependence of thermal expansion coefficient for several of the candidate mirror materials. In Figure 3-2 the variation of thermal conductivity with temperature for fused silica, beryllium and aluminum are described.

Figure 3-1 shows that while ULE and CER-VIT have negligible thermal expansion behavior at 20°C (300°K), fused silica has near zero coefficient at 190°K, beryllium at 40°K and aluminum at 15°K.

Thus no clear tradeoff exists for the choice of a mirror material expected to perform over a range of temperatures.

Summarizing materials criteria, it is apparent from an analysis of physical properties that no single material is outstanding in all categories. A compromise must be made among weight, stiffness, thermal performance and allied characteristics.

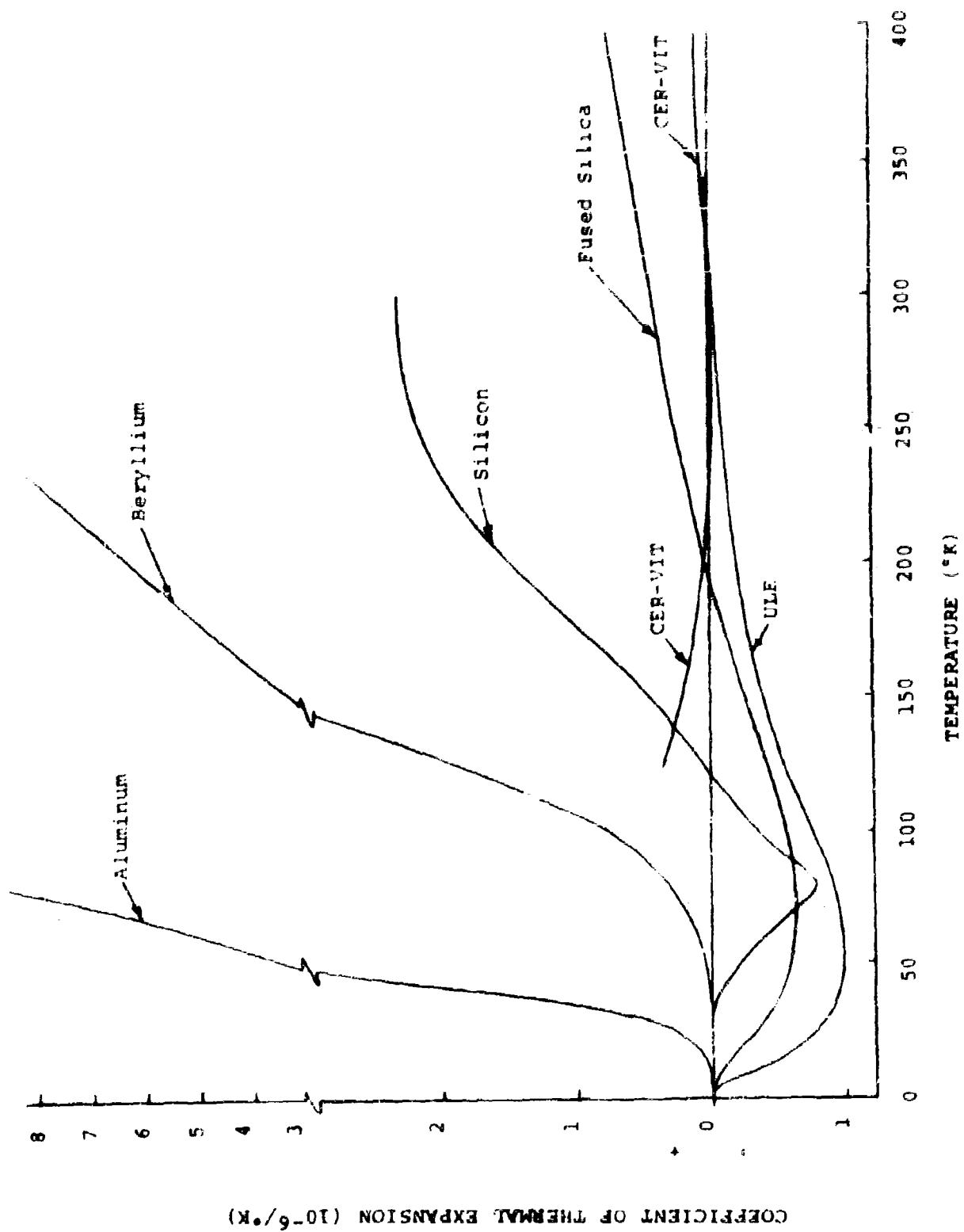


FIGURE 3-1 THERMAL DEPENDENCE OF THE COEFFICIENT OF THERMAL EXPANSION

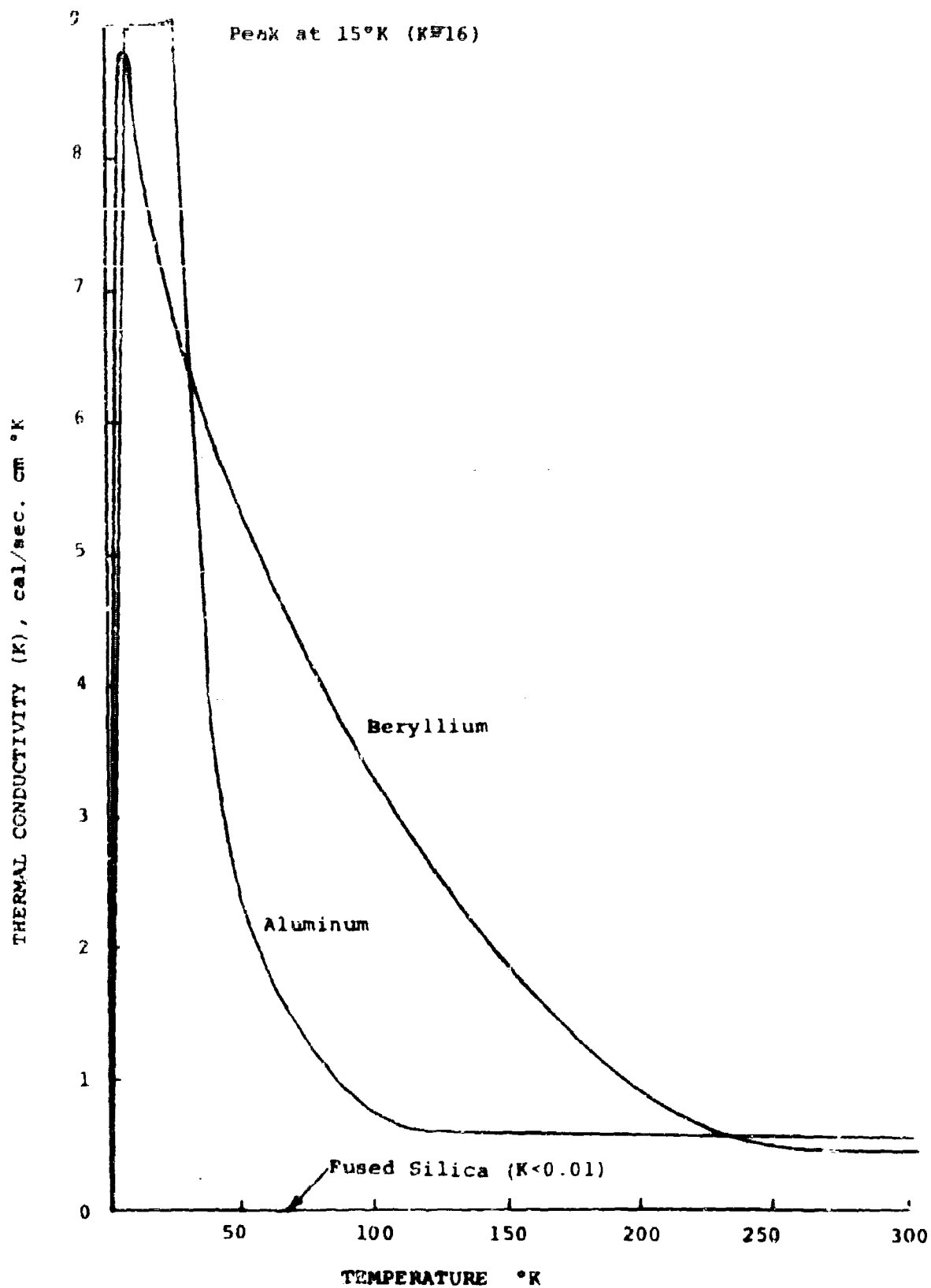


FIGURE 3-2 DEPENDENCE OF THERMAL CONDUCTIVITY ON TEMPERATURE

SECTION 4.0

MIRROR DEVELOPMENT AND OPTICAL CHARACTERIZATION

4.1 POLISHING OF MIRRORS

4.1.1 Beryllium Mirrors

Previous investigations⁽⁴⁾ have shown that the customary electroless nickel coatings used on beryllium mirrors were not necessary and in actuality were deleterious to optimum optical performance. Subsequently, a process⁽⁹⁾ for directly polishing beryllium surfaces was developed. Because of the inherent toxicity of beryllium, special precautions must be taken in the working of this material. When the polishing process is carried out as prescribed, the health and safety of personnel are not endangered.

Bare polished beryllium mirrors of up to 13 inches in diameter have been processed. Mirrors flat to $\lambda^*/10$ peak-to-peak, over 90% of aperture have been processed, as well as spheres accurate to $\lambda/5$. Surface quality is dependent on chemistry, oxide concentration and porosity. With proper combination of material characteristics, surface quality according to MIL-O-13830A is 80/50, or better.

The variation of mirror reflectance with oxide concentration and porosity was given earlier.⁽⁵⁾ In summary, the surface of a beryllium mirror should be fully dense and have a minimum of oxide inclusions. Beryllium mirrors with the best surface qualities were made of SP, P-20 (or the equivalent S-200-E), and the Be-Cr alloy of P-50.

* λ = wavelength = 0.6328 μm

The P-50 material is reported to have oxide concentrations ranging from 0.5 to 4.0 weight percent, however, its optical behavior resembles that of the low (0.5 - 2.0%) oxide materials. This may be due to a very fine dispersion of the oxide.

During this contractual period, a feasibility program was conducted to evaluate methods for manufacturing large mirror structures by joining together smaller component parts. Two joining methods were undertaken at BMI. These were a) brazing with an Al-Si filler and b) diffusion bonding.

Commercially manufactured mirrors were procured, polished and evaluated with respect to their thermal stability. These mirrors were then sent to BMI for joining (see Section 6.4). Mirrors were then returned to Perkin-Elmer where they were repolished and re-evaluated (see Section 5.5).

Four joined samples were processed; two were brazed and two were joined by diffusion bonding. One of the brazed samples failed during polishing. Failure occurred at the bond interface and it appeared as if the filler did not wet the beryllium matrix. The second brazed mirror was successfully polished with no joint visible to the unaided eye. Interferograms of this sample are shown in Section 5.5.

The two diffusion bonded mirror specimens exhibit a zone (approximately 5 mm wide) at the joint interface which could not be removed by prolonged polishing. This zone is softer than the beryllium matrix and is probably related to the Kirkendall porosity resulting from diffusion. The influence of this zone on thermal stability is almost insignificant as shown by interferograms in Section 5.5.

The importance of a "hard, polishable" surface of electroless nickel on beryllium is apparently insignificant. Although beryllium hardness is typically 250 Knoop and that of annealed electroless nickel is 700 Knoop, surface quality is primarily dictated by the polishing method and the lapping compounds used, rather than relative hardness. (7) The quality of polished surfaces of selected beryllium grades compares favorably with the best polished nickel coated mirrors.

Electroless nickel coatings on beryllium are generally undesirable (4,7) because of the thermally induced relaxation of high residual plating stresses, as well as the large mismatch ($2 \times 10^{-6}/^{\circ}\text{C}$) in thermal expansion between nickel and beryllium.

A nickel coating does provide useful corrosion protection to the non-optical surfaces of beryllium although feasible alternatives do exist such as anodizing and organic coatings.

4.1.2 Dielectric Mirrors

In this category of materials are included the oxide based materials which form glassy or glass-ceramic solids. Typical materials under investigation in this program are the glassy solids of fused silica and ULE, and glass-ceramics such as CER-VIT.

These materials can be prepared to high optical standards, but suffer to some degree from structural imperfections inherent in glass melts. Examples of these defects have been presented and the resulting effect on the polishing and stability of optical components has been described earlier (4,7).

4.2 OPTICAL CHARACTERIZATION

4.2.1 Polished Bare Beryllium Surfaces

As reported earlier (8,9) methods have been developed for directly preparing optical surfaces on beryllium substrates without an intermediate polishable coating.

It has also been recommended (4,9) (refer to Section 5.0) that all mirror surfaces should be chemically etched prior to optical polishing to remove surface regions previously damaged by machining, grinding, etc. Table 4-1 lists a recommended treatment for mirrors. Presence of "twins", a crystallographic transformation which occurs when applied stress exceeds a threshold quantity, can be detected by optical microscopy. (4) This inspection of the optical surface can therefore be used for quality control purposes.

4.2.2 Anodized Beryllium Surfaces

Studies were undertaken of anodized surfaces on beryllium to determine selected optical and mechanical characteristics. Through a literature search, an anodizing treatment was chosen on the basis of adherence and process control. The process parameters indicated in Table 4-2 were derived from a series of experiments designed to optimize anodized coating adherence and optical scatter.

TABLE 4-1

PERKIN-ELMER TREATMENT OF
BERYLLIUM MIRRORS

1. Rough Machine - allow 0.25 mm per side minimum over finish size
2. Etch - remove 0.25 mm per side in a solution of:
 - 27 cc H_2SO_4 (96%)
 - 410 cc H_3PO_4 (85%)
 - 70 g CrO_3
 - 130 cc H_2O
3. Stress Relieve
 - a. Heat slowly (300°C per hour maximum) to $900^\circ \pm 10^\circ C$ for one hour in vacuum.
 - b. Cool at 60°C per hour maximum to 200°C (vacuum not needed below 350°C).
 - c. Still air cool from 200°C to room temperature.
4. Finish Machine - in critical areas only if absolutely necessary.
5. Stabilize
 - a. Heat at uniform rate in 30 minutes to 100°C and hold 15 minutes.
 - b. Cool at uniform rate in 30 minutes to room temperature and hold 15 minutes.
 - c. Cool at uniform rate in 30 minutes to -75°C and hold 15 minutes.
 - d. Heat at uniform rate in 30 minutes to room temperature and hold 15 minutes.
 - e. Repeat steps 5a through 5d until 5 cycles have been completed.
6. Optical Polish

NOTE: At no time must the beryllium piece be allowed to undergo any thermal shock, i.e. $> 200^\circ C$ per hour.

Best Available Copy

TABLE 4-2

BERYLLIUM ANODIZING PARAMETERS

BATH COMPOSITION:	200 Grams CrO_3 1 Liter 10% HNO_3 (Volume %)
TEMPERATURE:	$26^\circ \pm 2^\circ\text{C}$
ELECTRODES:	6.3 mm spacing between two plane parallel beryllium cathodes and the sample (equal anode and cathode area).
VOLTAGE:	4.1 vdc (at electrodes)
CURRENT DENSITY:	0.03 amp/cm^2
TIME:	30 min.

Best Available Copy

It was found that properly anodized beryllium surfaces had highly unusual optical characteristics. In summary of this study, it was noted that an optically polished mirror having hemispherically scattered optical energy of 9.8%, had only 0.2% scatter after the anodizing treatment.

Reflectance of a) an optically polished beryllium surface, and b) an anodized beryllium surface are shown in Figure 4-1. Typically a high quality polished beryllium surface has reflectance of 49% over the visible spectrum, 90% at $\lambda=3\mu\text{m}$ and 95% at $\lambda=10\mu\text{m}$. Reflectivity dependence of bare beryllium upon residual porosity and BeO impurity content has previously been reported. (5,9) The anodized surface shown in Figure 4-1 has very low reflectivity (but low scatter, as described above). Reflectance is approximately 2% over the visible spectrum and reaches a maximum of 6% at $\lambda=7\mu\text{m}$ before decreasing to 5% at $\lambda=10\mu\text{m}$.

Since the total incident energy on a surface is either reflected (r), scattered (s), or absorbed (a), this normalized expression is valid:

$$1 = r + s + a$$

Since the (r) and (s) terms have been quantified, at least at $\lambda=0.63\mu\text{m}$, then it can be said that the absorption (at $\lambda=0.63\mu\text{m}$) is 0.98. The spectral absorptivity and emissivity (ϵ) are synonymous; therefore emissivity is also 0.98. A survey of other high emissivity materials shows that these anodized surfaces have a very high, if not the highest, reported emissivity. Since scatter probably follows a Rayleigh distribution law ($s \propto \lambda^{-4}$), then $\epsilon=1-r$ and (ϵ) can be obtained directly from Figure 4-1.

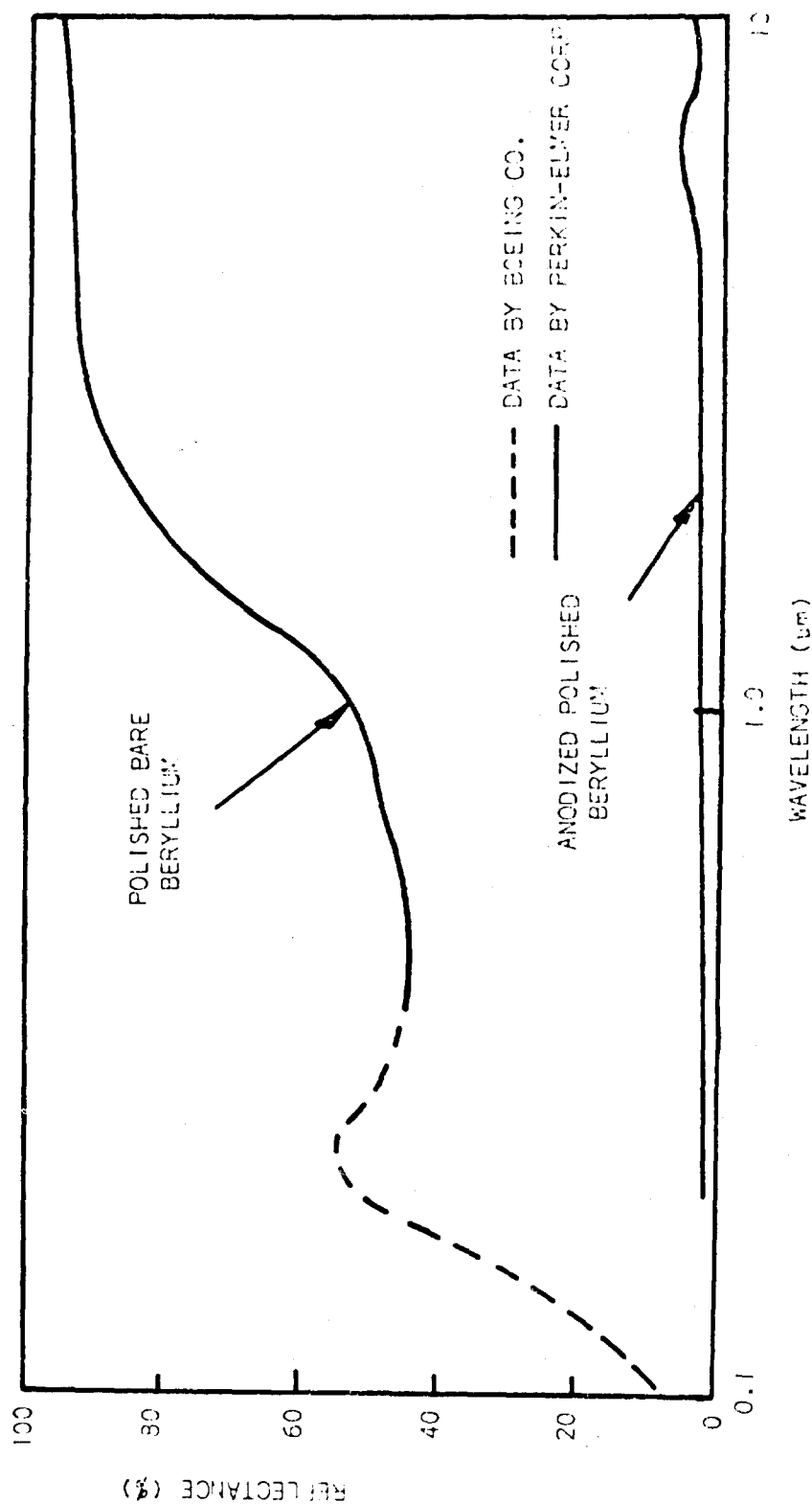


FIGURE 4-1 Reflectance of Polished Beryllium, Bare and Anodized.

On the basis of the low scatter of an anodized beryllium mirror, it is recommended that further studies be made of anodized coatings. The expansion of surface crystallographic cells during anodization apparently seals a number of voids, pores, etc., which act as light traps and scattering centers. By investigating anodizing process parameters in detail, it is believed that a thin (0.1 μ m) glass-like mirror surface coating can be developed on metal optics.

The main advantage of an anodized beryllium surface concerns its low scatter and high absorptance. This attractive combination of properties characterizes a "black mirror", i.e. a surface which absorbs a maximum of incident radiation and reflects the remainder, with scatter being negligible. A black mirror is very important as a baffling material in critical optical systems with low signal-to-noise ratios. Black mirror baffles made of anodized beryllium would then absorb 98% of incident visible energy and specularly reflect approximately 2%. A baffling design using multiple reflections would essentially completely attenuate unwanted rays.

Because of its high emissivity, the anodized beryllium surfaces can also be used advantageously for thermal control purposes in optical systems.

4.2.3 Optical Coatings on Mirror Surfaces

Development of improved adherence and spectral performance of high reflectance optical coatings have been described. (4,7,8)

Three optical quality high reflectance coatings which have been evaluated are:

- a) dielectric protected silver;
- b) dielectric protected aluminum; and
- c) VHR (a very high reflectance multilayer dielectric coating)

Absorption bands were present in the visible region for the early VHR coatings. Modifications in multilayer coating design were successful in shifting these bands to lower wavelengths. A flat response at maximum reflectance has been obtained from 0.43 μm to about 0.90 μm . Durability of these coatings is sufficient to pass the "scotch tape" adherence test.

The effect of glow discharge treatment on mirror substrate, cleaning and surface smoothing has been described. (7) Data indicate that prior to aluminizing a surface, a 10 minute glow discharge treatment in 100 millitorr pressure He or Ar at 2 KV and 100 ma produce a minimum scatter surface. Scatter decreases with lower aluminum film deposition rates. Deposition rates of 1-6 A/sec. are recommended.

Scattered energies at $\lambda=0.63 \mu\text{m}$ for several forms of polished, glow-discharged, and optically coated mirrors are listed in Table 4-3. The relatively high scatter (2-4%) of the dielectric-protected aluminum and silvered mirrors are values taken from single mirrors. It is felt that considerable reduction in scatter can be achieved with additional development work. For example, scatter of the VHR coatings was 4.1% several months ago, but is only 1.0% at present. If scatter of beryllium mirrors follows the Rayleigh distribution law ($S \sim \lambda^{-4}$), scatter at $\lambda=10 \mu\text{m}$ approaches 0.0001%.

TABLE 4-3
TOTAL HEMISPHERICALLY SCATTERED ENERGY FROM OPTICAL SURFACES
(Measured at $\lambda=0.63\mu\text{m}$)

MATERIALS	SCATTERED ENERGY %		
	Polished Bare Beryllium	Polished Kanigen-Coated Beryllium	Polished Glass
As Polished	0.20-1.2	0.16-1.00	0.02-0.10
Aluminized (No Protective Coating)	0.9-1.40		0.1-0.2
VHR Coating	1.0-1.5	2.05	0.2-1.0
Ag+SiO ₂ Coating	4.3	2.7	1.0
Al+SiO ₂ Coating	2.0	2.7	0.5

Small scratches, streaks and other polishing artifacts on optical surfaces probably cause scatter at longer wavelengths to deviate from the Rayleigh distribution law. It is therefore expected that the spectral distribution of scatter would reach a terminal level at long wavelengths instead of decreasing by λ^{-4} .

To date no extensive study of spectral and spatial distributions of scatter have been conducted as a function of optical surface quality.

SECTION 5.0

MIRROR TESTING AND MEASUREMENTS

5.1 GENERAL

In this program, mirror materials are evaluated in several ways. The dimensional instabilities of all materials are measured as functions of time, temperature, and thermal cycling. Homogeneity is evaluated by birefringence, isotropy in thermal expansion coefficient, and by X-ray diffraction. Residual stresses are measured interferometrically.

5.2 THE PRECISION SURFACE INTERFEROMETER

Dimensional stability is measured with the Precision Surface Interferometer.⁽¹⁰⁾ Twelve circular flat mirrors 107 mm diameter by 3.2 to 51 mm thick are evaluated simultaneously. Regular evaluations of mirror surfaces by high precision interferometry in a closely controlled thermal environment ($26.5^{\circ}\text{C} \pm 0.05^{\circ}\text{C}$) over an eight week interval provide information on long term, i.e. temporal, dimensional stability. By programming temperature changes (e.g. $26^{\circ}\text{C} - 35^{\circ}\text{C}$) in the interferometer, and analyzing interferograms taken at each temperature extreme, the thermal dimensional stability of each mirror is determined. Finally, all mirror specimens are interferometrically evaluated before and after 10 and 100 thermal cycles ($23.8^{\circ}\text{C} - 35.0^{\circ}\text{C}$, 100 minute period). Reduction of this data yields thermal cycle figure stability.

During this contractual period, four Test Programs of the above described sequence were scheduled. These Programs are identified as Test Programs VII, VIII, IX, and X. The Test Programs were carried out as follows:

<u>Program</u>	<u>Start Date</u>	<u>End Date</u>
VII	12 March, 1969	9 May, 1969
VIII	20 June, 1969	15 August, 1969
IX	18 November, 1969	13 January, 1970

Because of sample scheduling problems, the start of Program IX was delayed, and Program X was subsequently cancelled.

The interferometric test data of these recent test programs have been reduced by numerical methods⁽¹¹⁾. Instabilities are described in terms of the rms (root mean squared) change in surface figure as calculated from over 200 data points in the 107 mm diameter field of the mirror. The rms changes accompanying changes in time, temperature and thermal cycling have been reported for Test Programs VII and VIII.⁽⁷⁾ The rms changes accompanying time and temperature for specimens of Test Programs VII, VIII, and IX are listed in Tables 5-1, 5-2 and 5.3. Contour maps of the surface changes of these mirror samples and a complete description of results have also been presented^(4,7,8)

In these Tables, temporal instability data are given in units of λ rms per year for a wavelength (λ) of 0.63 μ m. Data obtained over an eight week test period are linearly extrapolated to a one year period. With the present detection sensitivity of 0.002 λ (rms), the

TABLE 5-1

MEASUREMENT DATA FROM TEST PROGRAM VII

SPEC. NO.	MAT'L/SUPPL'R/PROCESS ⁺	DIMENSIONAL STABILITY			
		TEMPORAL (rms)/yr	THERMAL ⁺ (rms)	THERMAL CYCLE λ (rms)**	
				10 Cycles	100 Cycles
81	Fused Silica, GE 151	0.031	0.004	not	cycled
63	Glass-Ceramic, Corning 9623	0.013	0.002	0.002	0.002
67	Glass-Ceramic, Corning 9622	0.016	0.003	0.002	0.003
76	ULE, Corning 7971	0.021	0.003	0.003	-
77	ULE, Corning 7971	0.019	0.002	0.002	0.005
178	I-400, BBC, VHP	0.048	0.006	0.003	0.011
179	S-200-D, BBC, VHP	0.113	0.018	0.017	0.009
131	P-40, RMI, HIF, (HOLLOW)	0.058	0.009	0.016	0.007
164	P-50 + 5% Cu, SRI, PS	0.035	0.010	0.006	0.016
165	FP 1755 + 5% Cu, SRI, PS	0.029	0.005	0.006	0.009
186	P-50, SRI, PS	0.035	0.005	0.005	0.011
191	SP, SRI, PS, etched	0.059	0.008	0.004	0.024

* For 6.4°C Excursion In Precision Surface Interferometer.

** Thermal cycling: 23.8°C - 35.0°C, period of 100 minutes.

+ All SRI samples fabricated from reattritioned powder

TABLE 5-2

MEASUREMENT DATA FROM TEST PROGRAM VIII

SPEC. NO.	MAT'L/SUPPL'R/PROCESS	DIMENSIONAL STABILITY			
		TEMPORAL (rms)/yr	THERMAL (rms)*	THERMAL CYCLE (rms)**	
				10 Cycles	100 Cycles
81	Fused Silica, GE 151	0.015	0.003	Not	Cycled
128	Be, FP-1755, BMI, HIP, (HOLLOW)	0.025	0.021	0.024	0.012
162	Be, HP-50, KBI, VHP	0.041	0.029	0.003	0.005
170	Glass-Keramik, Heraeus-Schott	0.029	0.011	0.006	0.006
175	Be, SP, KBI, VHP	0.043	0.053	0.019	0.019
190	Be, P-50, SRI, PS ⁺	0.049	0.013	0.007	0.007
195	Be, SP, SRI, PS ⁺	0.037	0.025	0.010	0.004
206	Be, P-40, SRI, PS ⁺	0.119	0.009	0.004	0.007
207	Be, P-40, SRI, PS ⁺	0.037	0.016	0.012	0.009
208	CER-VIT, C-101, Owens-Ill.	0.037	0.004	0.006	0.006
209	CER-VIT C-101, Owens-Ill.	0.014	0.005	0.005	0.003
210	TZM, NASA	0.009	0.004	0.004	0.008

* For 8.5°C Excursion in Precision Surface Interferometer.

** Thermal Cycling: 23.8°C - 35.0°C, period of 100 minutes.

+ Fabricated from reattritioned powder.

TABLE 5-3

MEASUREMENT DATA FROM TEST PROGRAM IX

SPEC. NO.	MATERIAL/SUPPLIER/PROCESS	DIMENSIONAL STABILITY	
		TEMPORAL λ (RMS)/YR.	THERMAL ** λ (RMS)
21	Fused Silica, GE 151	0.015	0.005
217	SP-200-E, BMI, HIP +	0.035	0.018
219	SP, BMI, HIP*	++	0.019
221	SP, BMI, HIP +	0.053	0.031
222	P-40, BMI, HIP*	0.340	0.060
225	P-50, SRI, PS*	0.080	0.010
227	P-50 + 5 Cu, SRI, PS*	++	0.024
230	P-40, BMI, HIP	0.064	0.012
233	SP-200-E, BMI, HIP*	0.139	0.015
235	SP, BMI, HIP	0.055	0.016
236	P-50, BMI, HIP*	0.042	0.018
238	P-50, BMI, HIP	0.100	0.006

+ Machined arch lightweight configuration.

* Fabricated from reattritioned powder.

** For 8.6°C excursion in Precision Surface Interferometer

++ Intermittent malfunction of camera

Best Available Copy

minimum detectable temporal instability from an eight week test is $0.013 \lambda(\text{rms})/\text{year}$.

The data indicate that the best BMI fabricated beryllium samples are temporally stable to two times the limit of instrument detectability. The best pressureless sintered products produced by SRI and the best commercial products produced by vacuum hot pressing are temporally stable to factors of two and three respectively. The best dielectrics and the one TZM mirror evaluated are stable to approximately the limit of detection.

A fused silica mirror (Sample No. 81) has now been evaluated in four successive programs. The rate of temporal instability has gradually decreased to an apparant minimum as follows:

<u>Test Date</u>	<u>Instability Rate</u>	<u>Age of Specimen</u>
June to Aug., 1968	$0.045 \lambda(\text{rms})/\text{yr}$	1.0 year
March to May, 1969	$0.031 \lambda(\text{rms})/\text{yr}$	1.7 years
June to Aug., 1969	$0.015 \lambda(\text{rms})/\text{yr}$	2.0 years
Nov. 1969 to Jan., 1970	$0.015 \lambda(\text{rms})/\text{yr}$	2.5 years

It appears that this fused silica mirror has become progressively more stable with time. The few dielectric mirrors tested which were not stable to the limit of detectibility of the instrument (e.g., Nos. 170 and 208) should be retested in order to determine if they will behave in a similar manner. This behavior is most probably caused by the gradual relaxation of residual stresses induced in the manufacturing, cutting and optical polishing operations.

Among the beryllium mirrors, the BMI and SRI fabricated samples are somewhat, but not substantially more stable than their commercial counterparts. The four commercial mirrors, Nos. 162, 175, 178 & 179, however, have all been etched and annealed in order to produce the most stable condition attainable. The SRI samples are inherently stable because of the processing method. The BMI samples as fabricated, may not be in their most stable condition. The stabilization treatment used by BMI and described earlier⁽⁴⁾ includes a two hour heat treatment at 788°C. This may not be a high enough temperature for complete relaxation of all residual stresses in the body of the material. The high hardness reported in Section 3.0 for BMI samples may be an indication of the need for a more extensive annealing treatment.

The data from Program IX show no apparent difference in the temporal or thermal stabilities of BMI samples made from as-received and reattritioned powder. If the theory expressed in the previous paragraph is correct, relaxation of the small amount of residual stress remaining in the samples could be masking any differences that might be present. Further study is indicated to resolve this situation.

5.3 REAL TIME DIMENSIONAL STABILITY MEASUREMENTS

A number of samples are being evaluated at approximately six month intervals during storage at room temperature. These "real time" data are being used to determine the validity of the extrapolations from the temporal stability data of the Precision Surface Interferometer. Performance data are summarized in Table 5-4.

TABLE 5-4

REAL TIME DIMENSIONAL STABILITY DATA
OF MIRROR SURFACES

SAMPLE NO.	(a) (b) MATERIAL/SUPPLIER/PROCESS	TIME (Months)	FIGURE CHANGE (λ RMS)*		
			>0.02	0.02-0.01	<0.01
2	FP-1755, SRI, PS	9		x	
6	FP-1755 + 1 Fe, SRI, PS	6		x	
7	FP-1755 + 1 Fe, SRI, PS	6			x
14	FP-1755, BMI, HIP	10		x	
23	FP-17, SRI, PS	9		x	
31	BeO, BBC, SC	15			x
35	FP-17 + 5 Cu, SRI, PS	9		x	
36	FP-17 + 5 Cu, SRI, PS	12			x
40	P-50, SRI, PS	9		x	
42	SP-200-D, BMI, HIP	7			x
58	SP-200-E, BMI, HIP	7		x	
79	Fused Quartz, GE-124	12		x	
80	Fused Quartz, GE-125	12		x	
82	Fused Quartz, GE-204	12			x
83	Fused Quartz, GE-510	12			x
84	Fused Quartz, GE-(510 + 124)	12		x	
90	I-400, BBC VHP (Kanigen)	13	x		
91	S-200-D, BBC, VHP (Kanigen)	13	x		
93	SP-200-E, BMI, HIP (Hollow)	10	x		
146	Silicon, WPAFB, C	7		x	
160	S-200-C, BBC, VHP (Kanigen)	9		x	
164	P-50 + 5 Cu, SRI, PS	13		x	
165	FP-1755 + 5 Cu, SRI, PS	13		x	
177	Be, BBC, SC	12			x
191	SP, SRI, PS	11		x	
194	SP, SRI, PS	8			x
213	Low Oxide, KBI, SS	6			x
208	Cer-Vit, O-I, C-101	6			x
209	Cer-Vit, O-I, C-101	6			x
77	ULE, Corning Type 797-1	8			x

- (a) SRI Stanford Research Institute
 BMI Battelle Memorial Institute
 BBC Brush Beryllium Company
 GE General Electric
 WPAFB Wright-Patterson Air Force Base
 KBI Kawecki-Beryllco Industries, Inc.
 O-I Owens-Illinois, Inc.
- (b) PS Pressureless Sintering
 HIP Hot Isostatic Pressing
 SC Slip Casting
 C Casting
 - Spark Sintering

* $\lambda = 0.6328 \mu$

Best Available Copy

All the samples evaluated have optical figure changes of less than $\lambda/50$ rms for their respective time intervals, except for three samples. Two of the three were coated with electroless nickel coatings, and the third was a hollow sample. A third electroless nickel coated sample was stable to approximately $\lambda/60$ rms over a nine month period.

Of the thirty samples which were evaluated, twenty-seven were stable to $\lambda/50$ rms or better. Twelve of these were better than $\lambda/100$ rms. Of the five best beryllium samples, three were produced by SRI, one by BMI and one by spark sintering.

Two fused quartz samples (GE grades 204 and 510) were stable to $\lambda/100$ rms, as were two CER-VIT (C-101) and one ULE (Corning 7971) sample.

Additional measurements should be made of these and other samples to obtain real time temporal dimensional stability data. Such data would approximate changes likely to occur during in-service use at this same temperature.

5.4 THE DIMENSIONAL INTERFEROMETER

Linear dimensional stability of cubical samples as evaluated with the Dimensional Interferometer has been described earlier.⁽⁴⁾ Table 5-5 lists all data obtained to date. Seven SRI cubes have been optically polished for testing. Long term measurements on six of these were not possible, as poor surface quality (with respect to voids) did not allow prolonged tests. The late delivery of beryllium cubes produced by BMI precluded their evaluation in this program.

With only two exceptions, one of which was an electroless nickel coated cube, all beryllium samples appear to be stable to better than 5×10^{-7} mm/mm/week. In general, the dielectric cubes are more

TABLE 5-5

LINEAR DIMENSIONAL STABILITY DATA
FROM PRECISION DIMENSIONAL INTERFEROMETER

Materials/Suppliers	Test Time (hrs)	Test Date (Start)	Average (a) Dimensional Change ^(b) (10^{-8} cm/cm/wk)
#105, Fused Silica, GE 151	44.6	4/3/69	42
#121, Fused Silica, Corning 7940	48.0	8/4/69	8
#124, Fused Silica ^(d) Corning 7940	29.0	11/10/69	0
	40.8	12/5/69	13
	32.5	12/17/69	32
#50, ULE, Corning 7971	27.5	7/2/69	17
#102, CER-VIT, 0-1 C-101	32.0	6/24/69	42
	30.5	6/26/69	42
#109, Glass Type E-3, Ohara	53.0	10/29/69	27
#100, Be, S-200, BBC, Electroless Ni	75.0	9/68	45
	23.8	5/22/69	171
#166, Be, SP, KBI	25.8	5/7/69	74
	26.6	5/13/69	64
#167, Be, SP, KBI ^(c)	148.0	7/15/69	10
	(193)		(27)
#197, Be, HP-40, KBI	30.0	12/23/69	29
#204, Be, SP, SRI	(e)	9/15/69	(e)
#205, Be, P-40, SRI	31.5	8/28/69	31

(a) The average dimensional change is the slope of the best fit straight line through the data points.

(b) Sensitivity of measurements ± 0.1 MHz or 6×10^{-9} cm/cm for a 5 cm sample length.

(c) There was a 10 MHz change in 9 hours after 148 hours. Values in parentheses are for the complete run and include this change.

(d) Data are for three orthogonal directions.

(e) Due to poor surface, no data were obtained for this test.

stable than the beryllium cubes. The fused silica samples No. 121 and 124 are the most stable of all cubes tested. More data must be accumulated before more definite conclusions can be drawn.

5.5 THERMAL DIMENSIONAL STABILITY

Thermal dimensional stability data not previously reported are presented in Table 5-6 and were primarily collected from interferograms taken at 25°C and 80°C. The actual rms figure change was divided by the magnitude of the temperature change to give the change in surface expected per unit change in temperature. Detection sensitivity for thermal stability measurements is approximately $1 \times 10^{-4} \lambda(\text{rms})/^{\circ}\text{C}$.

Summarizing all thermal dimensional stability data to date, most dielectric materials are stable to the limits of measurement sensitivity. Beryllium exhibits a greater range of thermal instability. Among the best beryllium samples tested, there are some produced by each of the three major methods, i.e., pressureless sintering, hot isostatic pressing and vacuum hot pressing. The best commercial samples noted were, however, from the same billet as some highly unstable pieces.

Although the thermal instabilities in most of the better beryllium materials are greater than those in the dielectric samples, in an absolute sense these instabilities are still small. For example, many specimens qualify for diffraction limited performance ($0.02 \lambda_{\text{rms}}$) over a 25°C temperature excursion. A number of these, including samples No. 6, 13, 186, 201, 203, 206, 207, and 223, will remain within specification over a 50°C change.

TABLE 5-6
THERMAL DIMENSIONAL STABILITY

SAMPLE No.	MATERIAL/SUPPLIER/PROCESS	STABILITY ($10^{-3} \lambda_{rms}^{**}/^{\circ}C$)
160	Be, S-200-C, BBC, VHP, 254 mm ϕ , Electroless Ni Coated	2.5
180	Fused Silica, Heraeus, 280 mm ϕ , Sandwich Lightweight	2.14
186	Be, P-50, SRI, PS*	0.39
190	Be, P-50, SRI, PS*	0.64
194	Be, SP, SRI, PS*	0.71
195	Be, SP, SRI, PS*	0.52
198	Be, HP-50, KBI, VHP	3.7
199	Be, HP-40, KBI, VHP	2.12
200	Be, HP-40, KBI, VHP	1.67
201	Be, HP-40, KBI, VHP	<0.10
202	Be, HP-40, KBI, VHP	0.78
203	Be, HP-40, KBI, VHP	<0.10
206	Be, P-40, SRI, PS*	<0.10
207	Be, P-40, SRI, PS*	0.26
213	Be, Low Oxide, KBI, Spark Sintered	0.75
216	Oharax, Ohara, 295 mm ϕ	1.33
220	Be, SP, BMI, HIP	2.9
223	Be, P-40, BMI, HIP*	0.15
228	Be, P-50, SRI, PS, 254 mm ϕ *	0.45
239	Be, P-50, BMI, HIP*	1.05
259	Fused Silica, Schott, 248 mm ϕ , Ultralight-Weight	<0.10

* Fabricated from reattritioned powder
 ** $\lambda = 0.6328 \mu m$

There appears to be no correlation between thermal and temporal instabilities. The former behavior is primarily dependent on the method of manufacture, while the latter is related to thermal and mechanical history. The thermal behavior of a number of specific samples has been previously described in detail.^(4,7) The differences among samples from the same vacuum hot pressed billet were ascribed to differences in crystallographic texture. It has been shown^(4,7,13) that X-ray diffraction can be used successfully to detect crystallographic preferred orientation in beryllium. This work is summarized in Section 5.6.2.

It is clear that hydrostatic methods for producing beryllium mirrors are superior to the commercial method of vacuum hot pressing for producing thermally stable products. The re-attribution method of powder processing further improves the thermal stability of beryllium products. This method statistically improves the probability of obtaining random crystal orientation within the body of the material. But elimination of preferred orientation is not all that is required. The presence of residual stresses in mirrors can cause mirror instabilities. For example, the six identical commercially produced mirrors which were tested⁽⁴⁾ in a thermal cycle test underwent both elastic and plastic thermal deformations. The plastic deformations were due to relaxations of the residual stresses induced in machining. Once these stresses were relieved by etching away the damaged material, or by annealing, the thermal behavior reverted solely to the elastic deformation previously observed. Even after two of these samples were cut in half and re-joined by diffusion bonding, the elastic thermal instabilities still remained as before. Table 5-7 lists the plastic and elastic de-

TABLE 5-7
THERMAL STABILITY OF BERYLLIUM MIRRORS
BEFORE AND AFTER JOINING

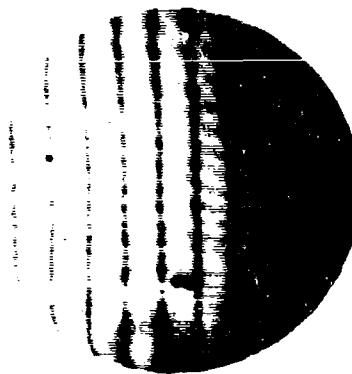
Sample No.	Condition	Thermal Stability, λ (peak-to-peak)	
		Plastic	Elastic
199	as machined	0.094	0.632
	etched	0.002	0.592
	brazed	0.210	0.585
200	as machined	0.104	0.597
	annealed	0.003	0.469
	diffusion-bonded	0.076	0.516
201	as machined	0.180	0.033
	annealed	0.016	0.027
	diffusion-bonded	0.002	0.017

formations observed in three of these mirrors (Nos. 199, 200, and 201) at three different times in their history. When tested as machined the plastic deformations in all three were substantial. After various treatments⁽⁷⁾ to determine the effects of etching and annealing on residual stresses in these pieces, their thermal stability was again documented, as indicated in Table 5-7 and also in Figures 5-1(a), 5-2(a) and 5-3(a). While the elastic changes in all three samples remained essentially the same, the plastic deformation was greatly reduced. After cutting and bonding, the elastic thermal behavior as shown in Figures 5-1(b) 5-2(b) and 5-3(b) was still as before, with additional plastic deformation due to bonding. The brazed sample (No. 199) processed at the lowest temperature had the highest plastic thermal instability. Sample No. 201, was optically ground less than Sample No. 200 and had the lowest plastic thermal instability. The bonding treatments are described in detail in Section 6.4.

Another example of residual stress causing thermal instability is that of Sample No. 228, a 254 mm diameter P-50 mirror. This sample was known to have almost random texture as measured by X-ray diffraction. It was not annealed after machining, but did have the surface damage from machining removed by chemical etching. The etching produced substantial pitting of the surface however, and considerable grinding was required during optical surface generation. The grinding introduced some residual stresses which relaxed during thermal evaluation of the piece. The mirror was initially cycled from 25°C to 80°C and returned. The elastic change was relatively small, but the plastic instability was approximately 1/5 peak-to-peak. A second similar cycle showed the elastic instability to be approximately



23°C

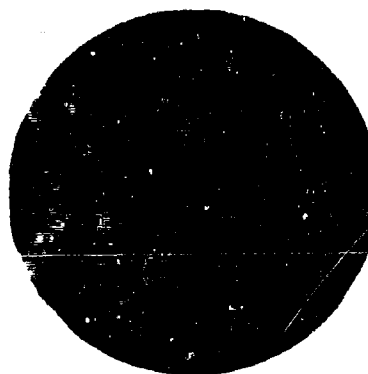


25°C



82°C

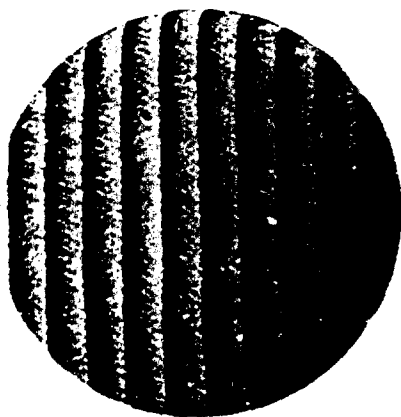
(a) Before



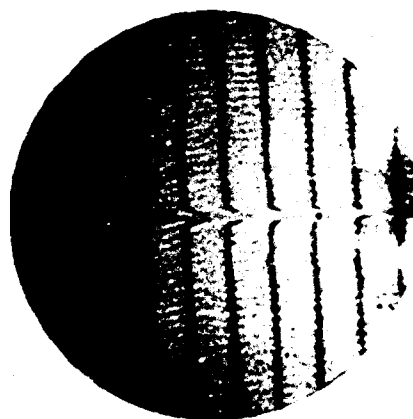
80°C

(b) After

FIGURE 5-1 THERMAL STABILITY OF SAMPLE NO. 199
BEFORE AND AFTER BRAZING



27°C

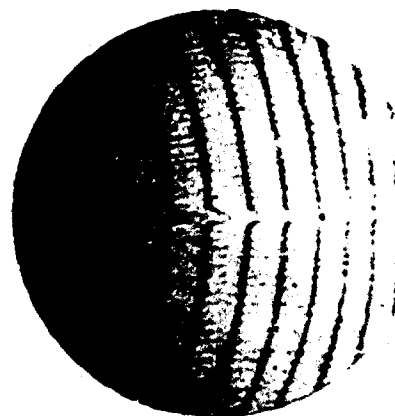


25°C



82°C

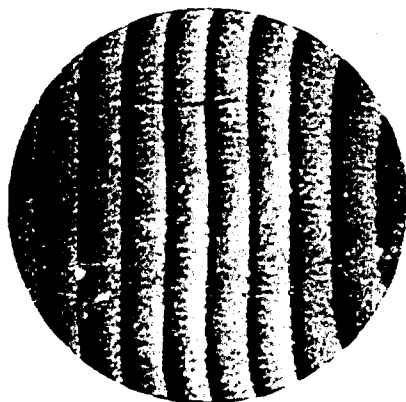
(a) Before



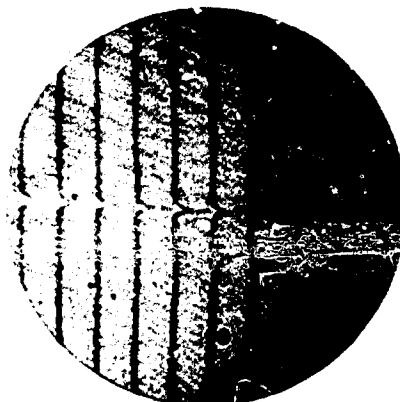
80°C

(b) After

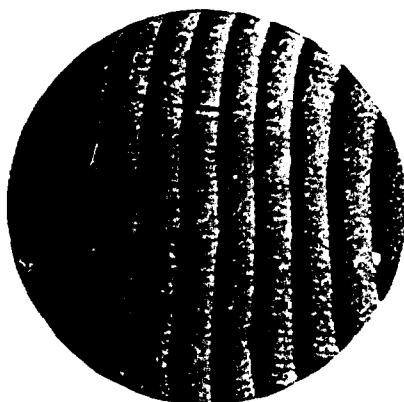
FIGURE 5-2 THERMAL STABILITY OF SAMPLE NO. 200
BEFORE AND AFTER DIFFUSION BONDING



27°C

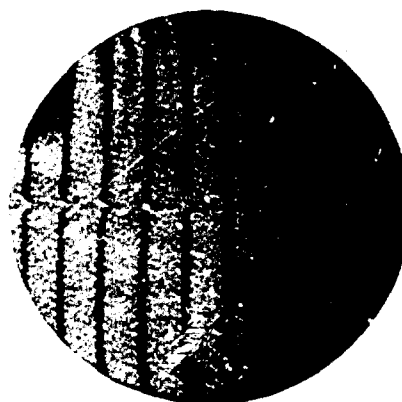


25°C



83°C

(a) Before



80°C

(b) After

FIGURE 5-3 THERMAL STABILITY OF SAMPLE NO. 201
BEFORE AND AFTER DIFFUSION BONDING

the same (1/10 peak-to-peak), while the plastic instability was immeasurable. Further cycling revealed no further plastic instability.

While residual stresses can be relaxed with thermal cycling they can also relax with time at a constant temperature. Maximum temporal stability therefore, as well as improved thermal stability, requires etching and annealing of machined surfaces, and a stabilization treatment for surfaces which have been optically ground.

Electroless nickel coatings have also been shown to be detrimental to thermal stability⁽⁷⁾ because of the stresses induced in plating and because of the thermal mismatch between coatings and beryllium substrates. The use of these coatings is not recommended on precision beryllium optical components.

5.6 HOMOGENEITY EVALUATIONS

Homogeneity tests are selected according to the material type under study. Birefringence evaluations of several dielectric materials were reported earlier^(7, 14). X-ray diffraction analyses of beryllium mirrors have also been reported.^(4, 7)

5.6.1 Thermal Expansion Coefficient Measurements

A Precision Vacuum Dilatometer⁽¹⁵⁾ capable of handling samples up to six inches long is being used to verify the hypothesis that some observed thermal instabilities may result from nonuniform thermal expansion in mirror substrates. Test data collected to date are listed in Table 5.5.

Previous reports^(4, 7) have discussed the 7.7% anisotropy in expansion coefficient of I-400 commercial beryllium (No. 172) and the 1.3% anisotropy in the pressureless-sintered sample (No. 28). Measurements of a ULE specimen (No. 52) parallel and perpendicular to striae show no significant anisotropy in thermal expansion.

It is known that reattritioning improves homogeneity in beryllium mirrors, but samples produced by this method have not yet been completely evaluated in the vacuum dilatometer due to sample scheduling problems.

5.6.2 X-Ray Diffraction

Because of a low atomic scattering factor and a resulting high transparency for X-rays, beryllium based products are

TABLE 5-8

THERMAL EXPANSION COEFFICIENTS
MEASUREMENT DATA

Sample No.	Material/Supplier	Temp. Range (°C)	Thermal Expansion (10 ⁻⁶ /°C)
-	CER-VIT, C-101	0-39	-0.040 ± 0.004
10	Be, SRI, FP-1755 + 1% Fe	2-38	11.14 ± 0.01
28	Be, SRI, FP-17, a+ b+	2-38	11.19 ± 0.01
		2-38	11.05 ± 0.01
205	Be, SRI, P-40	23-69	11.8 ± 0.1
171	Be, BBC 1-400, a* b**	2-38	10.36 ± 0.01
		2-38	10.42 ± 0.01
172	Be, BBC 1-400, a+ b+ c+	2-38	10.84 ± 0.01
		2-38	10.62 ± 0.01
		2-38	11.06 ± 0.01
167	Be, KBI, SP	2-38	10.36 ± 0.01
52	ULE ++	1-46	0.035 ± 0.021
	ULE +++	0-38	0.030 ± 0.008
105	Fused Silica, GE 151	2-39	0.604 ± 0.008
124	Fused Silica, Corning 7940	2-39	0.594 ± 0.007

+ Values are for mutually perpendicular cube axes,
unidentified with regard to pressing direction

* Parallel to pressing direction

** Perpendicular to pressing direction

++ Parallel to striae

+++ Perpendicular to striae

Best Available Copy

rarely monitored by diffraction. However, monitoring beryllium texture by X-ray diffraction has been refined and applied with considerable success. This method, though not totally conclusive due to the X-ray scattering characteristics of beryllium, has been instrumental in achieving a ten fold improvement of thermal stability in mirrors.

X-ray diffraction analyses have been reported and discussed at length^(4, 7, 13) In summary, the diffraction data, when compared with thermal dimensional stability data, indicate that the thermal behavior of beryllium mirrors is to a great extent a function of the crystallographic orientation. In general, the more random the texture, the better the thermal behavior will be. Samples produced by the hydrostatic methods of BMI and SRI using reattritioned powder exhibit greater thermal dimensional stabilities, and have been shown to have an essentially random texture.

It is suggested that precision beryllium components should be produced using a reattritioning method to achieve improved thermal performance. In addition, the X-ray diffraction method can be used for quality control in these applications.

5.7 RESIDUAL STRESS MEASUREMENTS

It has long been held that residual stresses in a material significantly contribute to both temporal and thermal dimensional instabilities. Stress-relief heat treatments, and the corresponding changes in mirror instabilities, have been previously documented for beryllium mirror specimens.⁽⁴⁾

In an effort to determine optimum stress relief treatment for beryllium, six identical discs (107 mm diameter, 13 mm thick) of vacuum hot pressed beryllium were procured from a commercial vendor.

The six discs were polished on one as-machined surface and thermally cycled from 26°C to 82°C and back to 26°C. All mirror surfaces at the elevated temperature were noticeably different from those at 26°C. Magnitudes of surface changes of the six "identical" mirror samples were noticeably different and some of the surface deformations observed at the elevated temperature were not totally recovered on returning to the initial temperature. This thermal behavior has been discussed in detail in a previous report.⁽⁴⁾

The plastic deformation detected after the thermal cycle test is most probably associated with deformations caused by thermal relaxation of residual stresses.

The magnitude and distribution of the residual stresses were then measured on the as-machined beryllium discs, as well as samples exposed to stress-relief heat treatments.

The data indicate that the residual stresses in as-machined parts are greater than was formerly realized. The stress-level at the surface approximates the engineering yield strength (1400 to 2500 kg/cm²), and decreases with increasing depth into the body. If the micro-yield strength of beryllium is 140 kg/cm² then a minimum of 0.25 mm must be removed for optimum stability. By measuring internal residual stresses in parts exposed to various stress relief heat treatments, it was concluded that heat treatments at 927°C and 982°C were apparently completely successful in removing residual stresses. A complete presentation of the data was included in the Third Quarterly Technical Report.⁽⁷⁾ As a result of these studies it is recommended that, for precision applications, beryllium be heat treated and etched (minimum 900°C/0.25 mm).

RADIATION DAMAGE

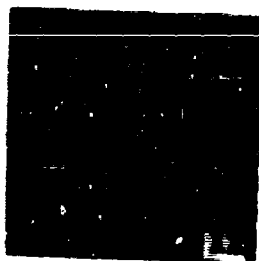
The radiation resistance of beryllium is well known as it is used as a moderator in nuclear reactors. Recently a number of mirror and optical coating test specimens prepared by Perkin-Elmer have been exposed to nuclear radiation in the Diesel Train experiment. In this experiment, the test specimens were exposed to the full spectrum of radiation from an underground nuclear detonation.

Optically polished bare beryllium surfaces survived the test with no change in surface geometry. Interferograms of two of these samples are shown in Figure 5-4, before and after exposure to the radiation. The only apparent change was the development of a thin oxide surface film from radiant heat and nuclear absorption phenomena. Dielectric samples, both bare and coated, were also exposed to the same environment but have not yet been received for evaluation.

Before Irradiation

After Irradiation

Sample
No. 12



Sample
No. 14

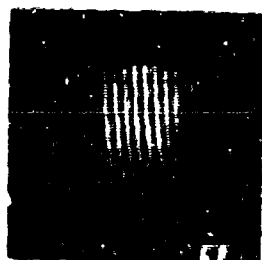


Figure 5-4 Radiation Effects on Beryllium Surfaces

SECTION 6.0

BATTELLE MEMORIAL INSTITUTE SUBCONTRACT

6.1 GENERAL

Under subcontract from this program, Battelle Memorial Institute has fabricated selected beryllium types and shapes by the hot isostatic pressing process. This effort was a continuation of prior development work conducted under the Optical Materials Study Program. In addition, feasibility tests have been conducted on methods for joining beryllium parts, the objective being to make large mosaic structures from smaller component parts.

This effort is described in the five tasks:

a. Task 1

Characterization of beryllium test specimens according to powder composition, particle size and distribution, processing parameters, density, homogeneity, and thermomechanical finishing treatments.

b. Task 2

Fabrication of three solid discs (107 mm diameter x 12.7 mm thickness) from three beryllium powder material types.

c. Task 3

Fabrication of three solid cubes (51 mm on a side) from three beryllium powder material types.

d. Task 4

Fabrication of three monolithic lightweight mirror^(5,8) blanks from three beryllium powder material types.

e. Task 5

Investigation of joining solid beryllium discs by diffusion bonding or brazing techniques.

Test specimens produced under Task 2 are similar in size and geometry to those obtained from other suppliers. These specimens are of a size amenable for surface stability evaluations in the Precision Surface Interferometer. Cubical specimens produced under Task 3 were to be tested in the Dimensional Interferometer.

6.2 TASK 1 - CHARACTERIZATION

A summary of the pertinent physical and chemical properties of the three powder types under study has been given in Section 3.0 of this report. A complete description of these powders has been reported earlier.⁽⁸⁾

The three techniques which were studied in an attempt to minimize preferred orientation were: a) reattritioning, b) consolidation of spherical powders, and c) radial compaction.

Berylum powder particles, as-received, have a plate-like geometry which causes nonrandom packing during processing of solid objects. The reattritioning method involves taking a cold hydropressed compact, made from as-received powder, and breaking it into small agglomerates. The agglomerate size of the reattritioned powder is 10 to 100 times the original powder size. This powder is then processed in the normal way as described earlier.⁽⁴⁾ Initial reattritioning was done by lathe turning and hand grinding. The current method involves cutting, crushing and ball-milling.

The use of spherical powders should eliminate nonrandom packing of the powders. This technique proved undesirable for several reasons. Grain size tended to be very large and the oxide tends to accumulate at the sphere surface. Density proved to be much less than theoretical, and so a high processing temperature would be necessary to achieve full density.

The third technique used to minimize preferred orientation is applicable only to the fabrication of disc specimens and is therefore limited in usefulness. This technique utilizes variable thickness rubber bags to counteract the orientation developed during vibratory packing by radially directing the particle motion during cold pressing. The texture of the resulting disc was not random, but was less pronounced than that resulting from normal compaction. These last two attempts at minimizing preferred orientation were discussed in detail in an earlier report.⁽⁸⁾

The effect of reattritioning in reducing preferred orientation was evaluated by X-ray diffraction and has been presented in detail in the Third Quarterly Report of this contract.⁽⁷⁾ Although some basal plane texture exists in the reattritioned, hot-isopressed material, the degree of texturing is significantly less than that in the as-received samples. Orientation effects in the machined arch lightweight configuration were relatively greater at the locations where the face was thinnest.

A total of thirty-nine specimens have been prepared under this program. Process parameters and the disposition of these specimens have been presented elsewhere.⁽⁷⁾ A history of each of the thirty samples delivered to Perkin-Elmer is included in Table 6-1.

All of the P-50 specimens processed achieved densities of 99.0 to 99.7% of their theoretical value. The acceptable P-40 samples ranged from 97.4 to 99.8%. The two SP samples delivered had densities of 99.8 and 100% as determined by Battelle.

The processing steps for all samples which were detailed earlier⁽⁴⁾ are summarized as follows:

TABLE 6-1
BERYLLIUM SAMPLES PREPARED BY BMI

SAMPLE NO.		POWDER		SIZE (mm)	REMARKS ^(e)
P-E	BMI	TYPE	CONDITION ^(a)		
217	10	SP-200-E	AR	107 Ø × 25.4 ^(b)	P,S,AL,1,100
218	14	P-40	R	107 Ø × 12.7	P,NG
219	20	SP	R	107 Ø × 9.5	P,AL,T,100
220	29	SP	AR	107 Ø × 9.5	P,LT,TC,H
221	30	SP	AR	107 Ø × 25.4 ^(b)	P,AL,T,100
222	31	P-40	R	107 Ø × 12.7	P,AL,T,100
223	38	P-40	R	107 Ø × 12.7	P,LT,TC,H
224	39	P-40	R	107 Ø × 25.4 ^{(b)(c)}	P,S,AL,S
230	1	P-40	AR	107 Ø × 12.7	P,AL,T,100
231	12	SP-200-E	R	107 Ø × 12.7	P,LT,H,NG
233	13	SP-200-E	R	107 Ø × 9.5	P,AL,T,100
235	3	SP	AR	107 Ø × 12.7	P,AL,T,100
236	17	P-50	R	107 Ø × 12.7	P,AL,T,100
237	23	P-40	AR	107 Ø × 12.7	P,H,NG
238	26	P-50	AR	107 Ø × 12.7	P,AL,T,100
239	34	P-50	R	107 Ø × 12.7	P,TC
240	37	P-50	R	107 Ø × 12.7	P,S,H
244	9	P-50	AR	51 × 51 × 51	
245	15	P-40	R	51 × 51 × 51	
246	18	P-50	R	51 × 51 × 51	
247	19	P-50	R	107 Ø × 25.4 ^(b)	P,S,AL,S
248	28	P-50	AR	107 Ø × 25.4 ^(b)	P,S,AL,S

TABLE 6-1 (CONT'D)

SAMPLE NO.		POWDER		SIZE (mm)	REMARKS ^(e)
P-E	BMI	TYPE	CONDITION ^(a)		
249	35	P-50	R	51 x 51 x 51	P
250	36	P-50	R	107 \emptyset x 25.4 ^(b)	
260	27	P-50	AR	51 x 51 x 51	
261	11	SP-200-E	AR	107 \emptyset x 25.4 ^(b)	NG
262	33	P-40	R	107 \emptyset x 12.7 ^(d)	
263	4	P-40	AR	107 \emptyset x 12.7	
264	5	P-50	AR	107 \emptyset x 12.7	

(a) AR Processed with "as received" powder
 R Processed with reattritioned powder

(b) Machined arch lightweight geometry

(c) Mirror shape machined from solid disc

(d) Disc shape machined from low density light weight

(e) All samples were etched and stabilized.

AL Flash aluminized

H Hardness measurement

LT Long term temporal stability evaluation (i.e. determined
 after periodic surface evaluation)

NG Sample not useable

P Optically polished

S Scatter measurement

T Temporal stability determined after 8 weeks in Precision
 Surface Interferometer as well as thermal stability.

TC Thermal stability determined between 26° and 82°C

100 Thermal cycle stability determined after 10 and 100 cycles
 between 24° and 35°C.

1. Containment in rubber bags
2. Vibratory compaction
3. Hydropressing
4. Rough machine to fit steel containers
5. Prepare mandrels for machined arch blanks
6. Fabricate steel containers and insert compacts
7. Outgas and seal containers
8. Hot-isopressing
9. Decanning
10. Final machining
11. Etch 0.25 mm minimum
12. Heat treat
13. Etch to final dimensions
14. Thermal cycle (stabilize)

6.3 FABRICATION

6.3.1 Task 2 - Solid Mirror Blanks

A total of sixteen solid discs out of the seventeen fabricated were sent to Perkin-Elmer. In addition, one unacceptable machined arch blank was machined into a disc. Of these seventeen discs, four are unacceptable because of cracks, fissures, voids and low density. Of the remaining thirteen, six are of as-received powder and seven were reattritioned. All but two (delivered in January 1970) of these samples have been polished.

6.3.2 Task 3 - Solid Beryllium Cubes

A total of six cubes out of the ten fabricated were sent to Perkin-Elmer. Of these six, three were reattritioned. There were four P-50 samples and one each of P-40 and SP. The latter two were of reattritioned and as-received powder respectively. None of

these cups have as yet been optically polished because of scheduling problems arising from the late delivery of all Battelle samples.

6.3.3 Task 4 - Lightweight Mirror Blanks

The design for lightweight mirrors is presented in Figure 6-1. The method chosen for producing these mirror blanks consisted of fabricating onto a mandrel having the proper contour. Mandrels were fabricated out of type 304 stainless steel, and barrier layers were used. Details of fabrication technology have been previously described. (4,8)

Seven lightweight mirror blanks of the twelve fabricated were sent to Perkin-Elmer. Of these, two were preliminary specimens of SP-200-E. Since the P-40 specimens did not densify adequately during hot isopressing, one specimen was fabricated by machining the specified shape from a thick solid disc. Of the remaining four lightweight mirror blanks, one is SP and three are of P-50 powder. Two of the P-50 samples and the P-40 sample are of re-attritioned powder. All but one have been polished.

6.4 Joining Studies

The second phase of this program was to evaluate the feasibility of fabricating beryllium mirrors by joining smaller segments of beryllium. Two techniques of joining were studied, viz., diffusion bonding by using either nickel or silver diffusion aids, and brazing.

6.4.1 Diffusion Bonding

6.4.1.1 Preparation of Preliminary Specimens

Thirty-six specimens were cut and ground from HP-40 rods supplied by the Perkin Elmer Corporation. The preparation of these

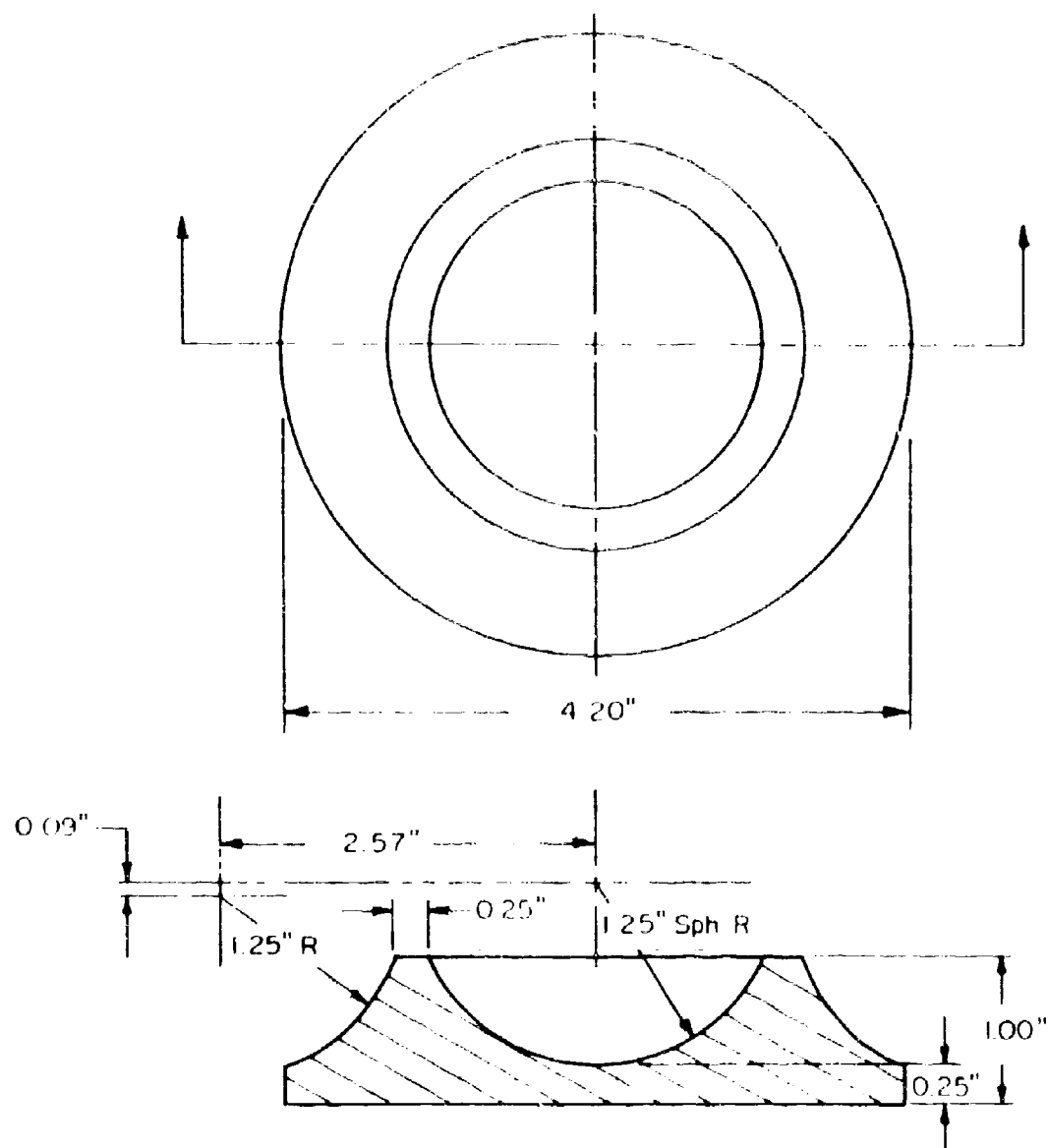


FIGURE 6-1 "MACHINED ARCH" LIGHTWEIGHT MIRROR DESIGN

specimens has been described in detail.⁽⁴⁾

The specimens were divided into three groups depending on surface finish: Group G - ground, Group E - etched, and Group L - lapped. Half of the specimens in each group were electrolytically coated with nickel to a thickness of 1.3 μm . The other specimens were coated on one face with vapor deposited (PVD) silver to a thickness of 0.6 to 1.3 μm .

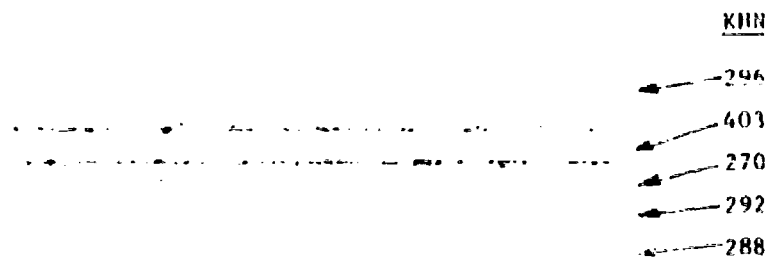
Four specimens out of each group, two coated with nickel and two with silver, or 12 total, were assembled in a column and wrapped with molybdenum foil. This first column of specimens was diffusion bonded by hot pressing in vacuum at 816°C and 28 kg/cm² for 4 hours. Metallographic analyses of the diffusion-bonded interfaces containing the nickel-diffusion aid showed the following:

- (1) Pellets were completely bonded across the interface and extensive beryllium grain growth occurred.
- (2) The Ni-Be diffusion zone was 30 - 50 μm thick.
- (3) Voids had formed in the beryllium immediately adjacent to the Ni-Be diffusion zone. Void formation was slightly less in those pellets which had ground-surface finishes.
- (4) Knoop hardness measurements (100-g load) indicated that the center of the Ni-Be diffusion zone was 100 units harder than the initial Be (403 KHN compared with 300 for unalloyed beryllium) indicating a nickel-beryllium solid solution.

Photomicrographs of a diffusion-bonded interface is shown in Figure 6-2.

Metallographic analysis of the interface containing the silver-diffusion aid showed:

- (1) Pellets with the silver coating on etched surfaces were only partially bonded across the interface. Pellets with either ground or polished surfaces achieved little or no bonding.



100X

a. As polished



100X

b. Polarized light

FIGURE 6-2 DIFFUSION BONDED NICKEL COATED BERYLLIUM

Beryllium surfaces were polished, electroplated with 1.3 μm of nickel, and hot pressed at 816°C for 4 hours at 28 kg/cm².

- (2) No grain growth occurred across the interface where bonding was evident.
- (3) The Ag-Be diffusion zone appeared to be very thin and could not be measured.
- (4) Void closure at the interface was incomplete.

Photomicrographs of a typical interface are shown in Figure 6-3. Pellets were easily separated at surfaces where no coating was applied.

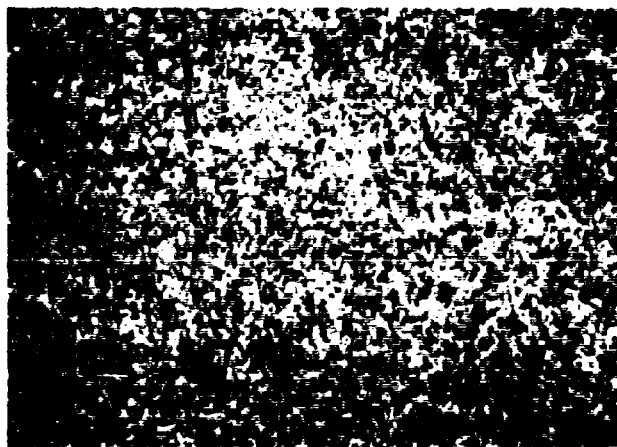
The second assembly of pellets contained only electroplated-nickel bonding aids. These pellets were uniaxially hot pressed at 677°C and 28 kg/cm² for 4 hours. Metallographic examination of the interfaces indicated that little or no bonding occurred between interfaces.

The third assembly of silver-coated beryllium pellets was uniaxially hot pressed at 815°C and 70 kg/cm² for 2 hours. Metallographic examination of the interfaces showed nearly complete bonding across every interface as shown in Figure 6-4. Most of the silver had diffused into the beryllium so that in one case, the interface was indistinguishable from the base material. A Be-Ag alloy phase was observed at the bond line. A few localized regions contained unalloyed silver. Grain growth across the interfaces was slight, but detectable. A very small amount of porosity existed at the bond line resulting from incomplete closure of interface voids. The most satisfactory silver-aided bond was achieved with pellets having polished surfaces.

To determine if satisfactory bonds can be achieved at lower pressures, the last series of hot-pressing experiments was conducted at 913°C and at a pressure of 7 kg/cm² for 1/2 hour. The bonding interfaces were examined metallographically and the joints were

100X

a. As polished



100X

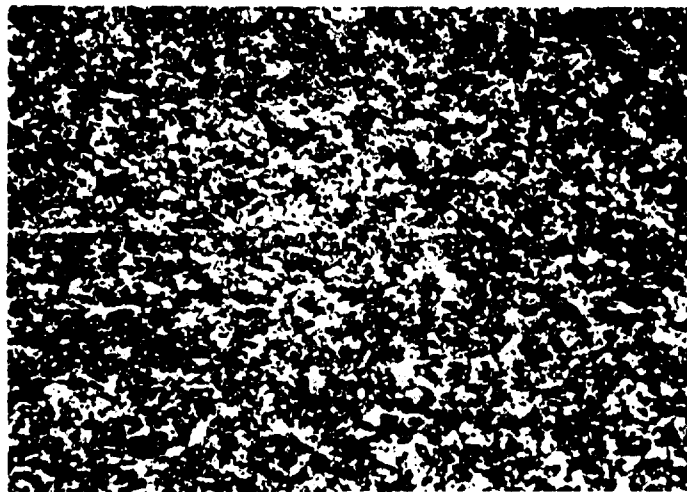
b. Polarized light

FIGURE 6-3 DIFFUSION BONDED SILVER COATED BERYLLIUM

Beryllium surfaces were etched in $\text{HF-HNO}_3\text{-H}_2\text{O}$ solution, coated by PVD with 0.6 μm . silver and hot pressed at 815°C for 4 hours at 28 kg/cm^2 .

100X

a. As polished



250X

b. Polarized light

FIGURE 6-4 DIFFUSION BONDED SILVER COATED BERYLLIUM

Beryllium surfaces were polished, coated by PVD with 0.6 μm of silver, and hot pressed at 815°C for 2 hours at 70 kg/cm^2 .

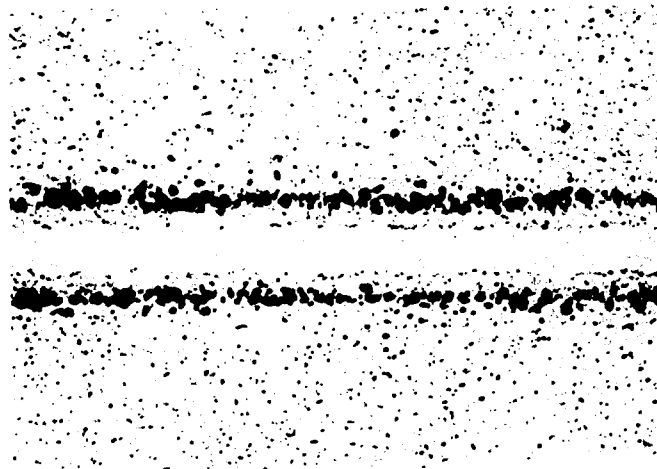
qualitatively mechanically tested. The nickel-coated interfaces were strongly bonded. The high bonding temperature (913°C) however, increased diffusion rates and vacancy migration, producing an excessive amount of porosity at the interface and extensive grain growth across the original surface. Figure 6-5 shows the microstructure of the bonded interface. For the silver coated samples, grain growth across the interface was negligible. Voids were observed, but these were small as shown in Figure 6-6. Silver coated surfaces did not produce strong bonds.

After this preliminary testing program, it was decided that all further diffusion bonding experiments would be carried out at 815°C, the nickel at 28 kg/cm² for four hours and the silver at 70 kg/cm² for two hours.

6.4.1.2 Preparation of Tensile Specimens

Conventional tensile tests were used to measure the tensile strength of nickel-aided and silver-aided diffusion bonds. Four tensile bars were prepared from beryllium rods 12.7 mm diameter by 102 mm long. Two of these rods were hot-pressed HP40, one was hot isopressed P-40, and the fourth was hot isopressed FP17 from last year's work. These rods were cut in half and the faces were ground. After preparing and cleaning the surfaces, the two hot-pressed HP40 rods were coated with 1.3 μ m of silver by vapor deposition. The hot-isopressed P40 and FP17 rods were coated with 0.6 μ m of electroplated nickel.

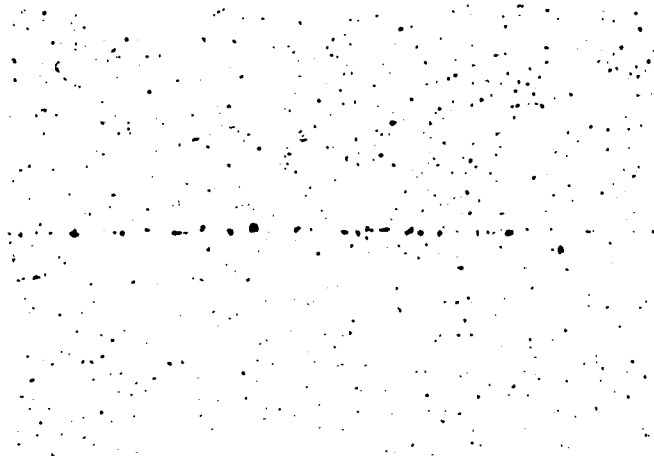
The thickness of the silver intermediate bonding aid on the tensile bars was slightly greater than that used earlier on pellets. This greater thickness was used to insure that sufficient silver



250X

FIGURE 6-5 DIFFUSION BONDED NICKEL COATED BERYLLIUM

Beryllium surfaces were polished, electroplated with 1.3 μm of nickel, and hot pressed at 913°C for 1/2 hour at 7 kg/cm².



250X

FIGURE 6-6 DIFFUSION BONDED SILVER COATED BERYLLIUM

Beryllium surfaces were polished, coated by PVD with 0.6 μm of silver, and hot pressed at 913°C for 1/2 hour at 7 kg/cm².

Best Available Copy

remained at the interface during bonding to achieve silver-to-silver contact and to prevent early depletion of silver from the interface. The thickness of the nickel intermediate bonding aid was reduced by 50 percent. By this reduction it was expected that complete bonding and adequate grain growth would still be obtained, but that the reaction zone and void volume due to the Kirkendall effect would be reduced.

The two nickel-coated bars and the two silver-coated tensile bars were bonded. In each experiment, the two bars having the same diffusion agent were bonded by placing the two bars end to end inside the hot-pressing fixture. Pressure was then applied to the other end of each tensile bar. Both nickel-coated tensile bars bonded satisfactorily using this type of pressing configuration. However, one silver-tensile specimen failed to bond, and a second cycle was required to form an adequate bond.

Tensile strengths of the diffusion-bonded joints were measured using an Instron Universal Testing Machine. The ultimate tensile strengths of the diffusion-bonded joints are summarized in Table 6-2. The nickel-aided diffusion-bonded specimens showed tensile strengths of 661 kg/cm² (FP17) and 696 kg/cm² (HIP P40). The silver-aided bonds had strengths of 2161 kg/cm² (1 cycle) and 585 kg/cm² (2 cycles).

6.4.1.3 Preparation of Disc Specimens

Two HP40 beryllium disks, Specimen Nos. 200 and 201, were sectioned as specified by Perkin Elmer and rejoined by diffusion bonding. Since only two disks were available, the nickel intermediate

TABLE 6-2 TENSILE STRENGTHS OF JOINED BERYLLIUM SPECIMENS

Joining Method	Temperature, °C	Pressure kg/cm ²	Time, hrs	Specimens		Bonding Agent	Tensile Strength, kg/cm ²
				Fabrication	Material No.		
Diffusion bonding	815	28	4	Hot iso-pressed	FP-17 16	0.6 μ m Ni	681
Diffusion bonding	815	28	4	Hot iso-pressed	P-40 27	0.6 μ m Ni	696
Diffusion bonding	815	70	2	Commercially hot pressed	HP-40 1	1.3 μ m Ag	2151 (a)
Diffusion bonding	815	70	2	Commercially hot pressed	HP-40 2	1.3 μ m Ag	585 (b)
Brazing	599	3.3	1	Commercially hot pressed	HP-40 1	Al-13 w/o Si foil	68
Brazing	599	3.3	1	Commercially hot pressed	HP-40 2	Al-13 w/o Si foil	82

(a) Loaded to 1406 kg/cm², unloaded to change load cells, and then reloaded to failure

(b) Required two temperature/pressure cycles for adequate bonding

bonding aid was used because of the lower pressure required. The bonding surfaces were ground.

Masking was applied up to about 1.6 mm of the interfaces to be bonded. Thus, a band about 3 mm wide at the interface was necessarily coated with 0.6 μ m of electroplated nickel to ensure that the bonding surface was completely coated.

The specimens were assembled in low-carbon steel containers with molybdenum foil barriers. Hot-gas pressure bonding was carried out at 815°C and 28 kg/cm² for four hours. The containers were then leached off. Although leaching the containers in nitric-acid solution introduced the risk of attacking the nickel at the bonding interface, care was taken to remove the specimens as soon as the containers were leached off to minimize any possible attack at the bonded interfaces.

Visual inspection of the diffusion-bonded disks indicated that (1) both disks were adequately bonded, (2) camber across the interfaces was negligible, (3) alignment of the two halves was maintained, and (4) the originally polished faces were replaced by dull, non-reflecting surfaces resembling an etched surface finish. The leaching solution appeared to have dissolved the nickel-rich interface slightly.

The bonded mirror blanks were sent to the Perkin Elmer Corporation for evaluation. The polishing operation was carried out successfully, but the interfaces were still visible after polishing. There were no differences in thermal stability before and after bonding.

6.4.2 Brazing

Brazing is a promising method for fabricating composite beryllium mirrors because low pressures can be used during joining.

This advantage becomes quite significant when considering scale-up for production of large mirrors.

6.4.2.1 Preparation of Preliminary Specimens

The aluminum-silicon alloy system was chosen for the filler alloy because of matching thermal expansion, relatively low brazing temperature and because neither aluminum nor silicon forms compounds with beryllium. The details of this selection process and the filler alloy preparation have been presented. (4,8)

Preparation of discs for brazing was identical to that for diffusion bonding, and has also been described previously. (4,8)

Components were brazed in a vacuum furnace using dead weight loading to provide 3.3 kg/cm^2 bonding pressure.

Table 6-1 summarizes the trial brazing experiments conducted in this program. It was decided initially to braze at high temperatures and for relatively long times to produce as much homogenization in the interfacial region as possible. This would minimize gradients of physical properties that might cause dimensional instability. A brazing temperature range of 577-666°C was selected for investigation because of the Al-Si eutectic temperature at 577°C, the Be-Al eutectic temperature at 645°C, and the melting temperature of pure Al-Si 660°C.

The initial brazes were made by using one molybdenum weight (1.6 kg/cm^2). Tests 1 through 7 displayed voids in the interface caused by dewetting of the Al-Si alloy on the Be surfaces. Interfacial voids were also produced at a lower brazing temperature of 621°C and a shorter brazing time of 30 minutes (No. 8).

Beginning with Braze Number 9, the heating procedure was modified to insure that the specimen, weights, and stainless steel platform were close to thermal equilibrium. Slow heating to the

TABLE 6-3 BRAZING EXPERIMENTS

Braze No.	Specimen Preparation	Heating Procedure	Time at Brazing Temp., min.	Pressure, kg/cm ²	Comments (a,b,c)
1	Ground surface; acetone and alcohol rinsed, air dried.	Rapidly to 649°C	50	1.6	Al-13Si foil, lightly etched in HNO ₃ HF solution. Excessive melting, numerous small voids
2	Ground to lapped surfaces, acetone and alcohol rinsed, air dried.	Rapidly to 649°C	50	1.6	
3	Lapped surfaces; acetone and alcohol rinsed, air dried.	Rapidly to 649°C	50	1.6	
4	Ground surface, etched.	Rapidly to 649°C	60	1.6	Foil lightly etched. Numerous small voids.
5	Ground and lapped surfaces, etched.	Rapidly to 649°C	60	1.6	Foil lightly etched. Numerous small voids.
6	Ground surfaces, hand polished on 600 grit paper, acetone rinsed.	Rapidly to 666°C	5	1.6	Foil rinsed with acetone. Small voids in braze. Edges of Br discs were rounded/hand polishing.
7	Ground surfaces, acetone rinsed.	Rapidly to 649°C	30	1.6	Foil rinsed with acetone. Large voids in braze.
8	Same as 7	Rapidly to 621°C	30	1.6	Foil rinsed with acetone. Numerous small voids.
9	Same as 7	Rapidly to 571°C slowly to	20	1.6	Foil rinsed with acetone. Foil did not melt.
10	Ground surfaces, lightly etched.	Rapidly to 566°C held 15 min., slowly to 580°C	60	1.6	Foil etched. Foil did not melt.
11	Same as 10	Rapidly to 571°C held 15 min., slowly to 582°C	60	1.6	Foil etched. Foil did not melt.
12	Same as 10	Rapidly to 580°C held 20 min., slowly to 593°C	10	1.6	Foil etched. Foil did not melt.
13	Same as 10	Slowly to 582°C held 20 min., slowly to 593°C	20	3.3	Foil etched. A few small voids, particularly near edges.
14	Same as 10	Slowly to 593°C held 20 min., slowly to 604°C	20	3.3	
15	Same as 10	Slowly to 571°C held 20 min., slowly to 582°C	20	3.3	Foil etched. Foil did not melt.

TABLE 6-3 (CONTINUED)

Braze No.	Specimen Preparation	Heating Procedure	Time at Brazing Temp., min.	Pressure, kg/cm ²	Comments (a,b,c)
16	Same as 10	Slowly to 568°C held 20 min., slowly to 577°C	20	3.3	Foil etched. Foil did not melt.
17	None (rerun of No. 16)	Slowly to 582°C held 20 min., slowly to 588°C	20	3.3	No voids. Slight gap near edges.
18	Ground surfaces, lightly etched.	Slowly to 582°C held 20 min., slowly to 590°C	20	3.3	Foil etched. Foil melted intermittently.
19	Ground to lapped surfaces, ground surface lightly etched.	Slowly to 582°C held 20 min., slowly to 590°C	20	3.3	Foil did not melt.
20	Al vapor deposited on one lapped surface, Si deposited on second lapped surface. Each coating 10-4 cm thick.	Slowly to 593°C held 20 min., slowly to 604°C	20	3.3	Braze opened in metallographic preparation. Alloy appeared only at joint edges.
21	None (rerun of No. 19)	Slowly to 588°C held 20 min., slowly to 599°C	20	3.3	Foil melted at edge but not in center
22	Ground surfaces, lightly etched, acetone rinsed.	Slowly to 593°C held 20 min., slowly to 599°C	60	3.3	Foil fully melted, no voids.

(a) All brazes were made in 2×10^{-6} torr vacuum.(b) All etching was done in a solution of 58 w/o $H_2O-40HNO_3-2HF$. Specimen surfaces were rinsed with acetone after etching.(c) All brazes except Number 20 were made using 12.7 μm - thick Al-13Si foil preplaced in the interface

brazing temperature or heating to a temperature below the brazing temperature, holding to attain equilibrium, and slow heating to the brazing temperature were used.

Brazing temperatures of 577°C to 593°C with a time of 20 minutes at temperature did not provide reproducible melting of the foil (Brazes 10-13, 15-19, and 21). The pressure used during brazing was raised to 3.3 kg/cm² because gaps were observed occasionally. Examination of braze number 17 indicated no melting at the edges. Metallographic examination showed islands of the primary solid solution of Al in Si within the Al-solid-solution matrix at both the center of the braze and in the unmelted foil indicating that very little melting of the foil occurred.

Brazes 18 through 21 were to determine a brazing temperature at which an adequate amount of melting would occur. Braze 21 was broken apart and indicated that thermal equilibrium had not been fully attained.

Braze Number 22 was made at 599°C for 60 minutes after equilibrating at 593°C for 20 minutes. Upon examination, it was observed that full melting had occurred across the interface. Therefore, these conditions were used for brazing tensile bars.

6.4.2.2 Preparation of Tensile Specimens

Commercially hot-pressed HP40 beryllium rods, 12.7 mm diameter by 48 mm long, were prepared for brazing by grinding the rod ends flat and parallel and lightly etching immediately before brazing as described previously. The Al-13Si foil was cleaned by wiping with alcohol. The specimens were loaded in compression to 3.3 kg/cm². The rods were brazed with the same procedure as that used for Braze Number 22.

Tensile tests were run in the same way as those used to evaluate diffusion-bonded joints. The tensile strengths of the two identically brazed rods were 82 kg/cm² and 68 kg/cm² as presented in Table 6-2.

6.4.2.3 Preparation of Disc Specimens

Mirrors supplied by Perkin Elmer (107 mm diameter by 11 mm thick) were cut along a prescribed diameter with a cutoff wheel.

The foil (Al-13 weight percent Si) was preplaced in the interface, the assembly was positioned in the vacuum furnace, and a load equivalent to 3.5 kg/cm² was applied. Specimen Number 198 was heated to 599°C in 60 minutes, held at the brazing temperature for 1 hour, and cooled to room temperature at a rate of 55°C per hour. The load was removed at about 540°C.

This brazing cycle did not fully melt the foil, so the brazing cycle was repeated at 604°C for 1 hour. Again, the foil that extended beyond the edges of the joint was not completely melted. A third brazing cycle at 610°C for 1 hour produced complete melting of the exposed foil. Specimen 199 was brazed in the same manner at 610°C. But again the exposed foil did not melt and so the mirror was rebrazed at 616°C for 1 hour. The exposed foil fully melted during this second brazing cycle. The two brazed mirrors were submitted to Perkin-Elmer for evaluation.

As described earlier, sample No. 198 came apart during the polishing operation, with little evidence of bonding. Sample No. 199 was polished successfully with the interface invisible to the unaided eye. The thermal behavior of this mirror was virtually the same as it was before brazing.

SECTION 7.0

SUMMARY

The Optical Materials Study Program is concerned with the mechanical, optical, and metallurgical characteristics of materials suitable for diffraction limited mirrors. The major portion of this study concerns the evaluation and development of beryllium (Be) mirrors.

Descriptions of the various mirror materials used in the program are outlined in Section 3.0. These include dielectric materials such as fused silica, glass-ceramics and low expansion glasses, and several beryllium-base products.

The most recent achievements in the preparation of bare Be optical surfaces are described. Material and processing specifications for high precision Be optical components are recommended. Brazed and diffusion bonded mirrors have been successfully polished and tested. An optically polished bare Be surface was anodized and results are presented which show low reflectivity and very low scatter. Recent improvements in optical coatings and scatter are reported.

Results of test programs carried out in the Precision Surface Interferometer are described in detail. The data indicate that most dielectric materials have rms thermal and temporal stabilities approaching 10^{-4} wave/°C and 10^{-2} wave/yr respectively. Hydrostatically prepared Be products are more stable than their commercial counterparts. Improvements have been observed in Be products through the use of a reattritioning process.

The reduction of temporal instability with time in a fused silica mirror is reported, and a mechanism is proposed. The relationship between residual stress and temporal stability in Be mirrors is also described.

Data are presented for mirrors evaluated at approximately six month intervals during storage at room temperature. Most mirrors have optical figure changes of less than 1/50 wave rms. Linear dimensional stability data of cubical samples evaluated in the Dimensional Interferometer are presented. Dielectric samples are generally more stable than beryllium samples.

Thermal instabilities are dependent on processing and also residual stress. Specific examples are presented. Homogeneity as determined by differences in thermal expansion coefficient and by X-ray diffraction is discussed. The use of reattritioning to improve homogeneity in Be products is recommended.

A summary of analyses of stress distribution in Be is presented. As a result of these studies, heat treating and etching of Be parts is recommended.

Data are presented which show no change in optically polished bare Be pieces exposed to nuclear radiation.

Under subcontract, Battelle Memorial Institute has fabricated Be samples for evaluation by Perkin-Elmer. A summary of all work on this program is presented. Fabrication techniques for the various geometries produced are described, as well as attempts to reduce preferred orientation in finished products. Detailed descriptions of brazing and diffusion bonding experiments are included.

Sectioned mirror samples were brazed with Al-Si foil and diffusion bonded using a nickel diffusion aid. Preliminary results show that fabrication of large mirror structures by joining small component parts is entirely feasible.

REFERENCES

1. R.M. Shemanski and R.E. Maringer, "Microstrain Characteristics of Isostatically Hot Pressed Beryllium", J. Less-Common Metals. 17, (1969), 25-45.
2. H.M. Brown, W.R. Goggin and J.W. Moberly, "Compacting and Pressureless Sintering of Beryllium Powders", Int'l J. Pwdr. Met., 5, 2, (1969)
3. John W. Moberly & Henry M. Brown, "On Fabricating Isotropic Beryllium", Int'l J. Pwdr. Met., 5, 4, 1969
4. Perkin-Elmer Report, OOD No. 13, August 1969, AD-858201-L.
5. Perkin-Elmer Report No. 9383, September 1968, AD-842356-L.
6. Private communication, J.W. Moberly, Stanford Research Institute.
7. Perkin-Elmer Report, OOD No. 29, November, 1969.
8. Perkin-Elmer, OOD No. 10, May 1969, AD-853936-L.
9. J.T. Bloxsom and J.B. Schroeder, "Preparation of Optical Surfaces on Beryllium", accepted for publication Appl. Opt.
10. R.G. Pilston and G. Steinberg, "Multiple-Wavelength Interferometry with Tunable Source", Appl. Optics, 8, 3, (March 1969).
11. R.A. Jones and L. Kadakia, "An Automated Interferogram Analysis Technique", Appl. Optics, 7, 8, (August 1968).
12. Perkin-Elmer Report No. 8903, August 1967, AD-819941-L.
13. William R. Goggin and John W. Moberly, "Thermal Dimensional Instabilities in Beryllium Mirrors", to be published.
14. Perkin-Elmer Report No. 9300, "Optical Materials Study Program," June, 1968
15. W.R. Goggin and R.A. Paquin, "High Precision Thermal Expansion Measurements Using a Frequency Stabilized Helium-Neon Laser," presented at the Laser Industry Conference, October, 1969.

Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author) Perkin-Elmer Corporation Optical Operations Division Main Avenue, Norwalk, Conn. 06852		2a. REPORT SECURITY CLASSIFICATION UNCLASSIFIED	
3. REPORT TITLE Optical Materials Study Program		2b. GROUP - - -	
4. DESCRIPTIVE NOTES (Type of report and inclusive dates) Final Technical Report (21 January 1969 to 20 January 1970)			
5. AUTHOR(S) (First name, middle initial, last name) Goggin, William R., Paquin, Roger A.			
6. REPORT DATE February, 1970		7a. TOTAL NO. OF PAGES 79	7b. NO. OF REFS 15
8a. CONTRACT OR GRANT NO. DAAH01-69-C-0950		9a. ORIGINATOR'S REPORT NUMBER(S) Optical Operations Division Engineering Report No. 41	
b. PROJECT NO. ARPA Order No. 885		9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) - - -	
c. d. - - -			
10. DISTRIBUTION STATEMENT This document may be further distributed by any holder only with specific prior approval of Director, Advanced Research Projects Agency, Washington, D.C. 20301			
11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY Advanced Research Projects Agency Dept. of Defense	
13. ABSTRACT Various materials are being investigated for use as diffraction limited mirror substrates. Included are dielectrics, such as fused silica, low expansion glasses and glass ceramics, and beryllium (Be) based materials. Material and processing specifications for Be optical components are developed and recommended. Sectioned Be mirrors were brazed and diffusion bonded and successfully polished and tested. Low reflectivity and very low scatter are reported for an anodized optically polished bare Be surface. Results of test programs of Precision Surface Interferometer are presented and discussed. Most dielectrics are temporally and thermally stable to approximately the limit of detectability. Hydrostatically prepared Be is more stable than the commercial counterparts and the reattritioning process has provided further improvement. The role of residual stress in thermal and temporal instabilities is presented. Data from Linear Dimensional Interferometer and Vacuum Dilatometer are presented. Details of brazing and diffusion bonding experiments for Be are included.			

Best Available Copy

DD FORM 1473

REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

UNCLASSIFIED

Security Classification

UNCLASSIFIED

Security Classification

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Glass Ceramics Fused Silica Beryllium Lightweight Mirrors Diffraction Limited Mirrors Thermal DIMensional Stability Temporal Dimensional Stability ULE Optical Properties CER-VIT Residual Stress in Beryllium Low Expansion Optical Materials Beryllium Fabricating & Processing X-ray Diffraction Reflectance of Optical Coatings Reattritioning Process in Beryllium Electroless Nickel Coatings Dielectric Mirrors Optical Scatter Vacuum Hot Pressing Hot Isostatic Pressing Pressureless Sintering						
Best Available Copy						

UNCLASSIFIED

Security Classification